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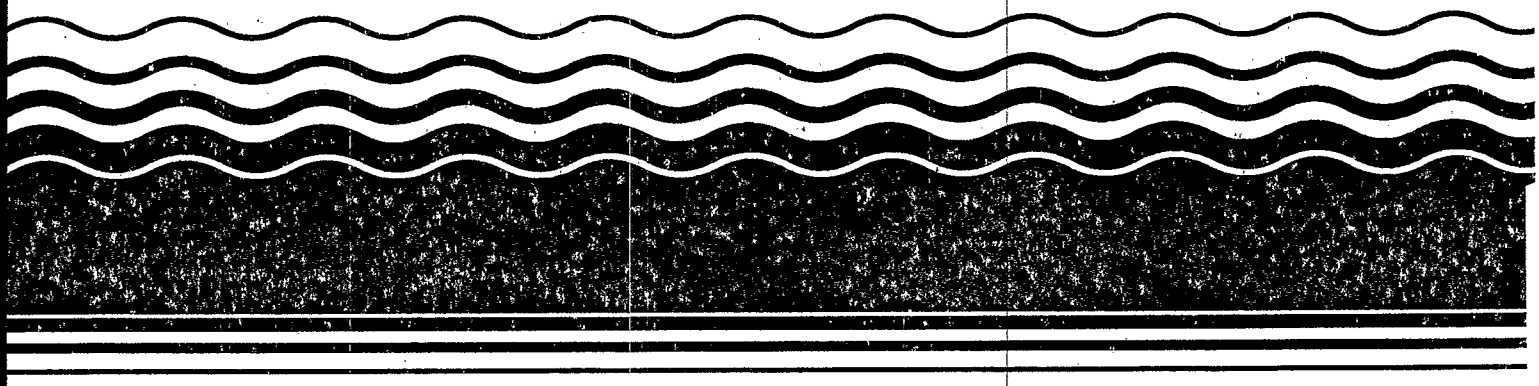
SUPERFUND METHOD FOR THE DETERMINATION OF RELEASABLE ASBESTOS IN SOILS AND BULK MATERIALS



SUPERFUND METHOD FOR THE DETERMINATION OF RELEASABLE ASBESTOS IN SOILS AND BULK MATERIALS

INTERIM VERSION

DISCLAIMER

Although this work was completed under contract to the U.S. Environmental Protection Agency, such support does not signify that the work or the conclusions drawn from the work necessarily reflect the views and policies of the Agency, nor does the mention of trade or commercial products constitute endorsement or recommendation for use.

A series of conceptual design figures are included in this method to assist users with their own design and construction of an appropriate dust generator for supporting this method. However, these figures are *not* to be construed as formal construction drawings and ICF Technology will accept *no* liability for their reference or use.

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1.0 INTRODUCTION

This is a sampling and analysis method for the determination of releasable asbestos in soils and bulk materials. Samples are collected in a manner suitable for providing representative measurements of the releasable fraction of asbestos in the matrix sampled, prepared using a dust generator, and analyzed by transmission electron microscopy (TEM). Guidelines for constructing the required dust generator are also included.

During dust generation, the respirable fraction of the dust generated from the sample is collected on filters. The filters are then weighed and the cumulative mass of dust collected is plotted against time to determine the rate of release of dust. Results are extrapolated to provide an estimate of the total mass of respirable dust in the original sample. The asbestos released during dust generation is either collected on a filter or in the suspension of a scrubber (or both), depending on the configuration under which the dust generator is operated. Filters may be prepared for TEM analysis by either a direct or an indirect transfer technique. Preparation of the scrubber suspension for TEM analysis is equivalent to an indirect transfer procedure. Depending on the intended use of the data, the results of asbestos analyses from this method may be reported either in terms of the number of structures per unit mass of respirable dust generated from the sample or the number of structures per unit mass of the original sample.

The method allows for the determination of the mineralogical type(s) of asbestos that is present in the sample and for distinguishing asbestos structures from non-asbestos structures. In this method, asbestos structures are characterized as fibers, bundles, clusters, or matrices and the length and width of each asbestos structure are measured. Although the method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it may be applicable to a wider range of studies.

As reported in this document, the method focuses on requirements for the collection, preparation, and analysis of samples obtained from individual locations. During a site investigation, samples will typically be collected from multiple locations that are arranged in an array designed to provide measurements suitable for deriving a representative (i.e. unbiased) estimate of the concentration of releasable asbestos over the sampled matrix as a whole. Thus, proper design of a comprehensive sampling strategy, which includes the detailed design of the array of sampling locations, is also critical to the success of an investigation. However, design of a sampling strategy is necessarily site specific and site-specific considerations are beyond the scope of this document. For further guidance on developing appropriate sampling strategies, see Berman and Chesson (undated).

This method has not yet been validated. Validation requires completion of a field study in which airborne exposure concentrations of asbestos caused by the release and transport of asbestos from soils or bulk materials (under specific conditions) are related to the bulk measurements of asbestos derived from this method. However, while such a study is in progress, this method has already been successfully tested in the laboratory. Such tests have demonstrated that the method is capable of achieving adequate sensitivity and precision to support risk assessment. The method also provides asbestos measurements that preserve the information on the sizes and shapes of asbestos structures that are required to assess risks. Thus, the principle features of the method are well enough established to allow it to be

employed in current field investigations. At the same time, until the formal validation study is completed, this should be considered an interim method.

NOTE

This document is intended to serve several audiences including site project managers, field sampling teams, data reviewers, and laboratory analysts. The document may be separated into segments so that individuals may focus on the sections of most interest to their particular roles on a project.

2.0 BACKGROUND

This method was developed specifically to satisfy the needs of the Superfund program including:

- the need to provide results suitable for supporting risk assessment;
- the need to be applicable to the types of asbestos-containing materials commonly encountered at Superfund sites; and
- the need to facilitate reproducibility within and between laboratories that may offer the method commercially.

An additional consideration addressed during the development of this method is the need to control sampling and analysis costs.

The first item listed above is what distinguishes sampling and analysis methods adapted for use in the Superfund program from methods used in other programs. This is because the *statutory requirements* of the Superfund program mandate that risk management decisions be based on risk assessment. Risk assessment requires that analytical data be relatable to health effects. Although the remaining requirements listed above are also important, the first item is a central feature of the Superfund program.

Because this method is designed for supporting risk assessment, the results of analyses from this method are intended to be used as inputs to release and transport models to predict airborne asbestos exposure concentrations (see Section 2.2). This is a very different objective from existing bulk asbestos methods, which are designed as qualitative tools for determining whether asbestos is present in a particular matrix in excess of a defined, regulatory limit. For such methods, the regulatory limits are defined operationally as functions of the methods themselves and, therefore, do not necessarily relate in any direct fashion to the potential for asbestos to be released and contribute to risk.

2.1 REQUIREMENTS FOR A METHOD DESIGNED TO SUPPORT RISK ASSESSMENT

A feasibility study (Berman 1990) was completed both to identify the requirements of a method for the determination of asbestos in soils and bulk materials that could be used to support risk assessment and to evaluate existing sampling and analysis technologies to determine whether such a method might be readily developed. Results of the feasibility study indicate that, to support risk assessment under Superfund, the method must:

- achieve sufficient analytical sensitivity to adequately measure asbestos over the entire range of concentrations that might potentially pose an unacceptable risk;
- provide adequate precision over the range of asbestos concentrations of interest;
- provide measurements of the complete range of the sizes and shapes of asbestos structures that are believed to contribute to health effects;

- provide measurements that are representative of the fraction of asbestos that is readily releasable[1] from the matrix of interest; and

- provide results reported in units that are amenable for use as inputs to fate and transport models that can be used to relate (bulk) source concentrations of asbestos to (airborne) exposure point concentrations of asbestos.

2.1.1 Sensitivity

Based on calculations presented in the feasibility study completed for this method (Berman 1990), asbestos concentrations in soil or a bulk environmental matrix that are on the order of $3x10^7$ long asbestos s/g_{solid} (i.e. 30 million asbestos structures longer than 5 μm per gram of solid) or $5x10^8$ total asbestos s/g_{solid} (i.e. 0.5 billion total asbestos structures per gram of solid) may potentially pose a risk exceeding 1×10^{-6}. This is based on evaluation of a series of scenarios in which asbestos is released from the solid matrix due to any of several types of disturbance (i.e. vehicular traffic on the surface, agricultural tilling, or natural weathering). Once released, asbestos is dispersed into the air, where exposure may occur. Note that, for a typical chrysotile matrix, the concentrations reported above for the two different size ranges of structures are expected to be approximately equivalent (i.e. concentrations observed for each of the two size ranges in a sample containing common chrysotile are expected to be approximately proportional to the two values given so that a sample observed to exceed one will also likely exceed the other). Therefore, either concentration might serve as an adequate target sensitivity for chrysotile. However, such may not be the case for all asbestos-containing matrices, particularly those containing amphibole asbestos.

To assure that negative results from a soil-bulk method do not mask potential problems, ideally, the analytical sensitivity for the method should be set at one tenth of the concentrations potentially capable of producing risks that exceed 1×10^{-6}. Therefore, the target analytical sensitivities for this method have been set at:

- $3x10^6$ long asbestos s/g_{solid} (i.e. 3 million asbestos structures longer than 5 μm per gram of solid); or

- $5x10^7$ total asbestos s/g_{solid} (i.e. 50 million total asbestos structures per gram of solid).

The analytical sensitivity is reported for the two different size ranges of asbestos structures to allow flexibility in the application of the method.

2.1.2 Precision

The only precision data currently available for this method is from the recently described pilot study (Berman et al. 1994a). To establish a reasonable goal for the precision of this method, a study of the precision achievable by commercial laboratories performing the method would be required. Because data from commercial laboratories are not available, the data from the

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[1] As used here, "readily releasable" means particles that have already been separated into respirable size and that are available in a pool of loose material that can be released directly during some type of disturbance. This is distinguished from particles that may be aggregated with others and that may be separated from the aggregate for future release.

pilot study (Berman et al. 1994a) were evaluated to provide a rough estimate of the level of precision potentially achievable once the method is commercialized (see Section 12.1.2). Results suggest that a relative percent difference of 50% should be easily achievable for sample splits performed by a *single* laboratory.

The USEPA has set informal precision guidelines for each of the analytical methods employed in the contract laboratory program (CLP), under which the majority of sample analyses have been conducted for the Superfund program. These goals tend to be defined as achievable relative percent differences for sample splits performed within a laboratory and they range between 10% and 35% for the analysis of inorganic chemicals in soils and up to 50% for the analysis of organic chemicals in soils. Therefore, given the above, a guideline of 50% for the relative percent difference between sample splits performed within a laboratory is proposed for this method.

2.1.3 Asbestos Characteristics

Based on what is known about the biological activity of asbestos (for a critical review of the extensive literature on this subject, see Berman and Crump 1989), if asbestos measurements are to be related to risk, it is necessary to characterize the sizes, shapes, and mineralogy of the asbestos structures in each sample. This involves enumeration of individual structures within certain size categories with particular emphasis on the longest and thinnest structures. Although the range of dimensions over which asbestos structures contribute to biological activity has yet to be precisely defined, this method is designed to provide a detailed characterization of structures encompassing the entire range of potential importance.

Results of the feasibility study for this method (Berman 1990) indicate that transmission electron microscopy (TEM) is the only analytical tool capable of characterizing asbestos structures over the entire range of sizes and shapes that potentially contribute to risk. Consequently, the asbestos derived from soil or bulk samples is analyzed using TEM in this method.

When evaluating detailed asbestos size characterizations, it is important to consider the effects of sample preparation. The dust generator incorporated into this method was developed because its use eliminates the need to employ other preparation techniques (such as crushing or grinding) that potentially alter the distribution of respirable asbestos structure sizes and shapes found in the sample. The dust generation employed in this method is a gentle process; it is expected to preserve the distribution of asbestos structure sizes and shapes that may be released to the air when asbestos-containing media are disturbed in the environment.

The size distribution of asbestos structures found in the dusts generated using this method may also vary depending on whether TEM specimen grids are prepared from the dust samples using a direct or an indirect transfer technique. Existing risk factors are based largely on studies incorporating the equivalent of direct transfer techniques,[2] while indirect transfer techniques are expected to provide increased precision (see, for example, Berman

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[2] Most of the epidemiology studies (from which estimates of asbestos potency are derived) employed either phase contrast microscopy (PCM) for the analysis of asbestos concentrations in work place air or converted other types of measurements to PCM equivalents (Berman and Crump 1989). PCM analyses are performed directly on sample filters after the filter material has been rendered transparent by a suitable solvent. Since the fibers are observed as originally deposited, this corresponds closely to a direct transfer technique for TEM analysis.

and Chatfield 1990). Hence, this method incorporates a procedure by which the majority of samples are prepared by an indirect technique with a subset prepared in tandem by a direct technique. This facilitates evaluation of the relationship between structure counts derived from samples prepared, respectively, by each technique.

2.1.4 Reporting Requirements

It is anticipated that soil or bulk samples to be analyzed using this method will typically be collected to determine the asbestos content of a potential source from which asbestos may be released (via a particular mechanism) and transported to the air (where exposure may occur). It is also expected that asbestos release and transport will be modeled and that one of the critical inputs to such models will be the concentration of releasable asbestos in the source matrix.

Results of the feasibility study for this method (Berman 1990) indicate that most of the available models that predict releases to the air from soil or some other bulk matrix (in association with specific release mechanisms) were designed to predict the release and transport of respirable dust. Only a very limited number of such models have actually been developed specifically for asbestos. The dust models may be used to predict asbestos release and transport with minimal modification, however, provided that the appropriate types of asbestos measurements are available. Such adaptations rely on the following assumptions:

- the rate of settling of respirable asbestos particles is no more rapid than the average settling rate for respirable dust; and
- the release of asbestos and the release of respirable dust from a source matrix are highly correlated (i.e. are proportional), at least over the long term.

The former assumption is expected to be true because fibers tend to settle more slowly in the air than spheres of comparable mass. The latter assumption appears reasonable because, if it were false, one would expect the source matrix to become either enriched or depleted in asbestos over time. However, such effects are not generally observed.

Many of the release models developed for respirable dust require the mass fraction of silt in the source matrix as an input parameter. For such models, substituting the fiber concentration of asbestos (per unit mass of source matrix) for the mass fraction of silt should allow the model to be used to predict asbestos release (with no additional modifications). Under such circumstances, a dimensional analysis of the model should indicate that the outputs would now be expressed in terms of the number of *asbestos structures* released from a defined area (or mass) of the source matrix per unit time (rather than the mass of *respirable dust* released from a defined area or mass of the source matrix per unit time). Such outputs are then typically combined with air dispersion models to predict airborne concentrations at locations (i.e. points of exposure) of interest.

As an alternative, it may be useful to multiply the respirable dust release rates (that are predicted by a model) by a factor representing the number of asbestos structures (of a size range of interest) per unit mass of respirable dust released from the sample. For certain models, this approach for converting a dust model to an asbestos release model may prove easier than the approach discussed above.

This method is designed to provide results that can be reported as the number of asbestos structures (of a size range of interest) per unit mass of source matrix with an option allowing the reporting of the number of asbestos structures per unit mass of the respirable dust released from the sample. The appropriate reporting option should be selected based on the specific models with which the measurements from this method are anticipated to be used for a specific project.

NOTE

This method may also be used to provide an independent (qualitative) check of the predictions of specific release models in some cases. In one of the intermediate steps of this method, the rate of release of respirable dust from the sample is determined. This can be compared to the rate of release predicted by a release model. The relative magnitude of the release rate observed using this method and the release rate predicted by a model should be consistent with that expected based on the relative aggressiveness of the type of disturbance applied to the sample during measurement using this method and the type of disturbance associated with the field activity (i.e. vehicular traffic, excavation, wind entrainment, etc.) represented by the model.

2.2 ASBESTOS CONTAINING MATERIALS TYPICALLY ENCOUNTERED AT SUPERFUND SITES

Asbestos containing materials commonly encountered at Superfund Sites include:

- natural rocks that contain asbestos;
- soils containing natural asbestos generated from weathered rock;
- soils containing asbestos introduced by transport from other locations;
- mine or mill tailings (i.e. fractured or depleted rock);
- discarded asbestos wastes, including (for example):
  - asbestos/cement pipe;
  - roofing materials;
  - insulation materials; and
- soils containing discarded asbestos wastes.

This method is designed specifically to handle the above materials (with the exception of unfractured rock[3]) and may be applied generally to samples of any unconsolidated or friable[4] matrix.

2.3 REQUIREMENTS FOR FACILITATING REPRODUCIBILITY BETWEEN LABORATORIES

Results of the feasibility study for this method (Berman 1990) indicate that, for a method for the determination of asbestos in soils and bulk materials to adequately facilitate reproducibility between laboratories that might offer the method commercially, the following requirements must be satisfied:

- preparation steps in the procedure have to be kept simple and must be standardized and documented sufficiently to allow technicians in different laboratories to perform them invariably (i.e. objectively); and
- the representativeness of a sample (i.e. the degree with which the sample retains the characteristics of the matrix from which it is derived) has to be preserved throughout all stages of handling, preparation, and sub-sampling that are incorporated into the method.

To address each of the above requirements, the ability to homogenize the sample has to be maintained throughout all stages of sample preparation and handling, and dust generation satisfies these requirements. In addition, dust generation is incorporated into this method as the means of extracting the respirable fraction of releasable asbestos from bulk samples in a manner that can be performed invariably; it is a mechanical process that can be controlled objectively by specifying the design and the operation of the equipment to be used for dust generation.

Use of a dust generator eliminates most of the sample manipulation steps typically performed manually (and subjectively) for other bulk asbestos methods. More importantly, it eliminates the need for sub-sampling of amounts smaller than approximately 100 g, which is a mass that is large enough to be sub-sampled reproducibly (by following specified procedures). A 100 g sample is also sufficiently large to retain representative characteristics of all components of a sampled matrix in which the particles are smaller than approximately 1 cm in diameter.

Results of the feasibility study (Berman 1990) indicate that the ability to homogenize an unconsolidated bulk sample (in preparation for sub-sampling) is a direct function of the largest particle in the distribution of component particles; to allow representative sub-sampling of such a matrix, the largest particle must represent no more than a few percent of the total mass of the sample. Therefore, because the final sub-sample to be extracted in this method is on the order of 100 g, the largest particles that can remain in the sample prior to sub

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[3] It is assumed that any asbestos imbedded in unfractured rock can be considered to be non-releasable. If there is a desire to evaluate the release of asbestos from the surface of such rock (or the potential for release of asbestos from the rock as it becomes fractured in the future due to aging or disturbance), conceivably, a sample of the rock might be crushed to particles no larger than 1 cm in diameter (see text) and a sample of the crushed rock might then be analyzed using this method. However, no formal protocol has been developed for this procedure at this time.

[4] As used here, the term *friable* is intended to mean any material that can be crushed or deformed with the hand with the attendant release of fibers.

sampling must be no larger than 2 or 3 g. Assuming a typical density for silica type materials (i.e. 2.6 g/cc), the largest particle that can be retained while allowing 100 g samples to remain representative of the sampled matrix is approximately 1 cm (3/8th inch) in diameter ($4/3\pi r^3$*density = 1.4 g). Consequently, this method incorporates a step in which samples are sieved to remove particles larger than 1 cm in diameter[5].

It must be emphasized that the particles larger than 1 cm in diameter that are removed from the sample by sieving in this method are *not* discarded. Rather, both the fraction passed by the sieve and the fraction retained by the sieve are weighed and the weights recorded so that the asbestos content measured can ultimately be reported as a function of the *total* mass of the initial sample (i.e. the asbestos concentration measured in the method is multiplied by the ratio of the total mass of the original sample collected and the mass of the fraction of the sample analyzed). The larger particles do not need to be carried through the entire analysis, however, because it is unlikely that significant amounts of the releasable asbestos in a sample reside in the coarse fraction.

2.4 COST CONSIDERATIONS

To be useful, it was determined that the cost of an individual analysis using this method would have to be competitive with other methods that might be used to derive comparable information.[6] In fact, once a laboratory invests in the construction of a dust generator to support the method and procures the required ancillary equipment, it is expected that the cost of analysis using this method will be very competitive with other soil or bulk methods (that might be designed to provide similar information). This is because use of the dust generator effectively concentrates asbestos from the sample (by removing the non-respirable component). Such concentration allows higher loadings on specimen grids so that smaller areas need to be scanned with the TEM to complete an analysis.

It is expected that sample preparation using the dust generator will cost approximately $400 (twice what it costs to complete the preparation of an air sample using an indirect transfer technique). This cost, however, is expected to be more than compensated by the corresponding reduction in cost for TEM scanning time, which is reduced due to the ability to scan more highly concentrated samples (see above). It is therefore expected that the total cost of an analysis using this method may run between $900 and $1,500, which should be competitive with any other soil or bulk method that is designed to provide comparable information. This cost is approximately 10 to 15% higher than that for the analysis of an air sample in which comparable structure size information is recorded.

Incorporated into this method (as an option) is a compositing procedure that can be used to reduce significantly the number of bulk analyses that might otherwise be required to adequately characterize a source matrix from which asbestos may be released (Section 8.3). Recognizing that airborne exposure due to emissions from a source matrix tend to be the result of average emissions over relatively large areas, compositing of samples is a

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5 The dust generator designed for this method best handles samples up to a maximum of approximately 80 g. However, the practical difference between 80 and 100 g samples, in terms of the maximum size of the particles that can be retained (while assuring the ability to homogenize the sample), is not significant.

6 This assumes analysis using TEM in which comparable size and mineralogy information is recorded; methods in which analysis is performed by polarized light microscopy (PLM) are not capable of providing information over the complete range of structure sizes and shapes that are believed to relate to risk.

particularly powerful tool that can be used in tandem with this method. By reducing the number of sample analyses required to adequately characterize a particular source, the cost of a particular investigation can be reduced correspondingly.

An additional cost saving measure incorporated into this method is completion of the early stages of sample preparation in the field. This simplifies the handling and preparation performed by the laboratory and limits the size (mass) of each sample that has to be stored or disposed by the laboratory.

Handling and preparation of bulk samples in the laboratory are dusty operations that require protective enclosures. The larger the sizes of the samples, the larger the protective enclosures required. Additionally, asbestos containing samples handled by a laboratory must be disposed as asbestos wastes. Clearly, the larger the mass of such samples handled by the laboratory, the greater the cost of disposal.

3.0 OVERVIEW OF METHOD

Samples are collected in the field according to a pre-defined sampling plan identifying the number of samples to be collected and the locations from which samples are to be collected. Procedures for designing such a plan are beyond the scope of this document but are reported elsewhere (see, for example, Berman and Chesson, undated).

Any of a variety of commercially available sampling equipment (i.e. trowels, shovels, augers, corers, etc.) may be used to collect samples for this method. However, they must have been specified in the pre-defined sampling plan based on the nature of the material being sampled and the depths over which samples are to be collected. Whatever sampling technique is employed, the *minimum* size sample to be collected at each location shall be 1 kg.

Once collected, each sample is brought to a central location for field preparation. Field preparation steps are listed in Figure 3-1 and discussed in detail in Chapter 8. Each sample is first weighed (Section 8.2.1). Then the sample is sieved using a screen with 3/8th in. (1 cm) openings to separate a coarse and fine fraction. The material placed on the sieve is worked with gloved hands to assure that all friable components pass through the screen (Section 8.2.2).

The coarse fraction, composed of material that is retained by the screen, is transferred to a bucket and weighed prior to discarding on site. The fine fraction is also weighed. As indicated in Figure 3-1, the fine fraction is then homogenized. The procedure recommended in this method for homogenization is repetitive splitting using a riffle splitter with the split halves of the sample being re-combined at the end of each split (Section 8.2.3). Studies indicate that five to seven iterations are typically sufficient to achieve adequate homogenization.

Once homogenized, the fine fraction is then sub-sampled using the riffle splitter (Figure 3-1). During sub-sampling, the one-half of the sample from one of the two receiving trays is discarded after each split (Section 8.2.3) and the second half of the sample is then re-split. The process is repeated until sub-samples weighing between 50 and 80 g are produced in each of the two receiving trays. The material in each tray is then transferred quantitatively to a sample bottle, packaged and shipped to the laboratory.

Sample handling, preparation, and analysis in the laboratory is depicted in Figure 3-2 and described in detail in Chapter 9. Once sub-samples weighing between 50 and 80 g are obtained, they can be separately prepared and analyzed (Section 9.2).

To prepare samples, as indicated in Figure 3-2, first load the sample into the tumbler of a dust generator. The design, construction, and operation of a dust generator suitable for use with this method is provided in appendix A. The sample is then conditioned by flowing humidity-controlled air through the tumbler and over the sample for several hours (Section 9.4.2).

Once the sample is conditioned, the tumbler of the dust generator is started and a sample run is initiated (Section 9.4.3). During each run, a series of filters is collected continuously from the top of one of the openings of the dust generator and these are weighed to plot the cumulative dust loss from the sample (Section 9.4.4 and the right side pathway of Figure 3-2).

FIGURE 3-1

SAMPLE COLLECTION AND FIELD PREPARATION

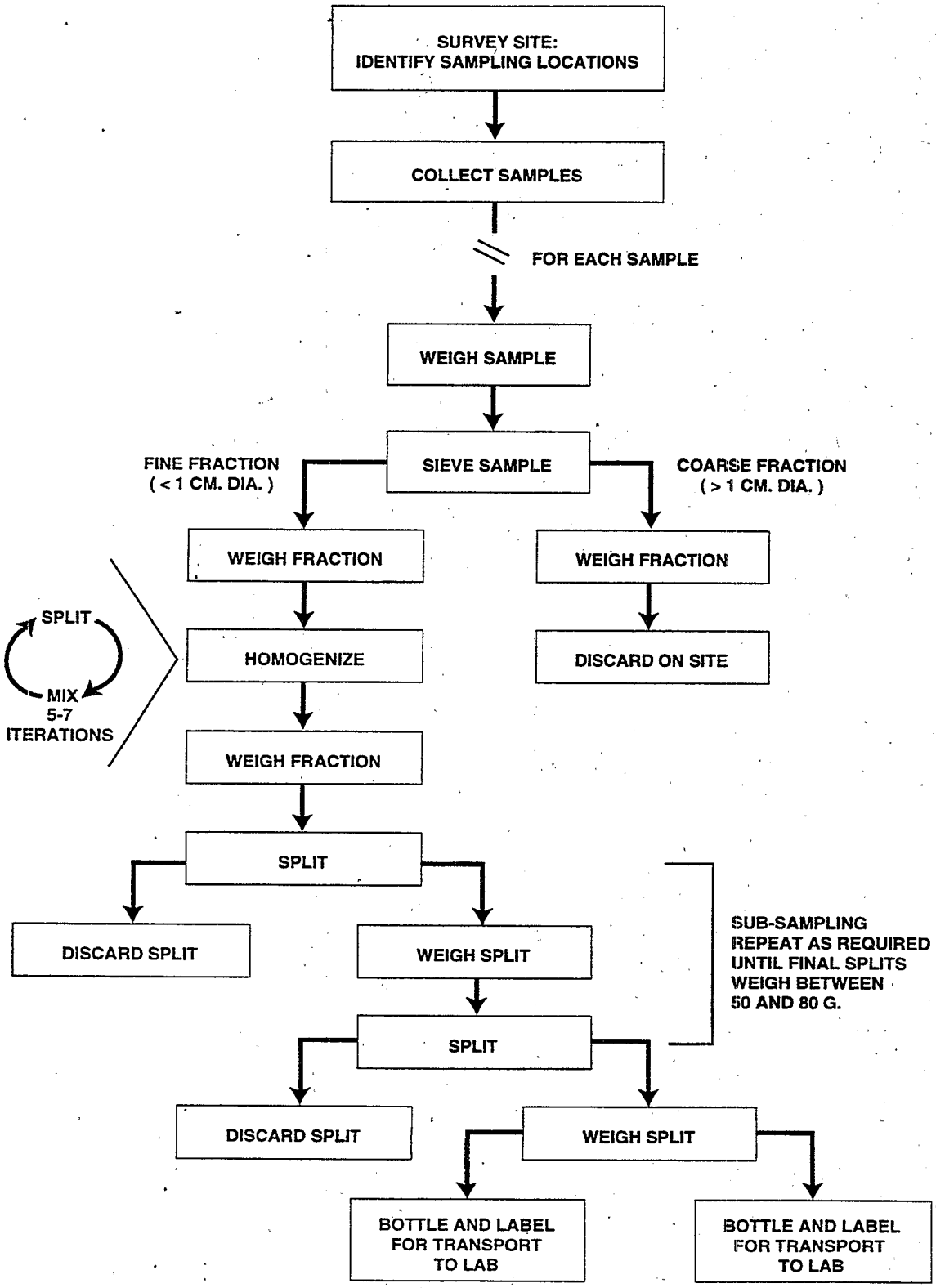
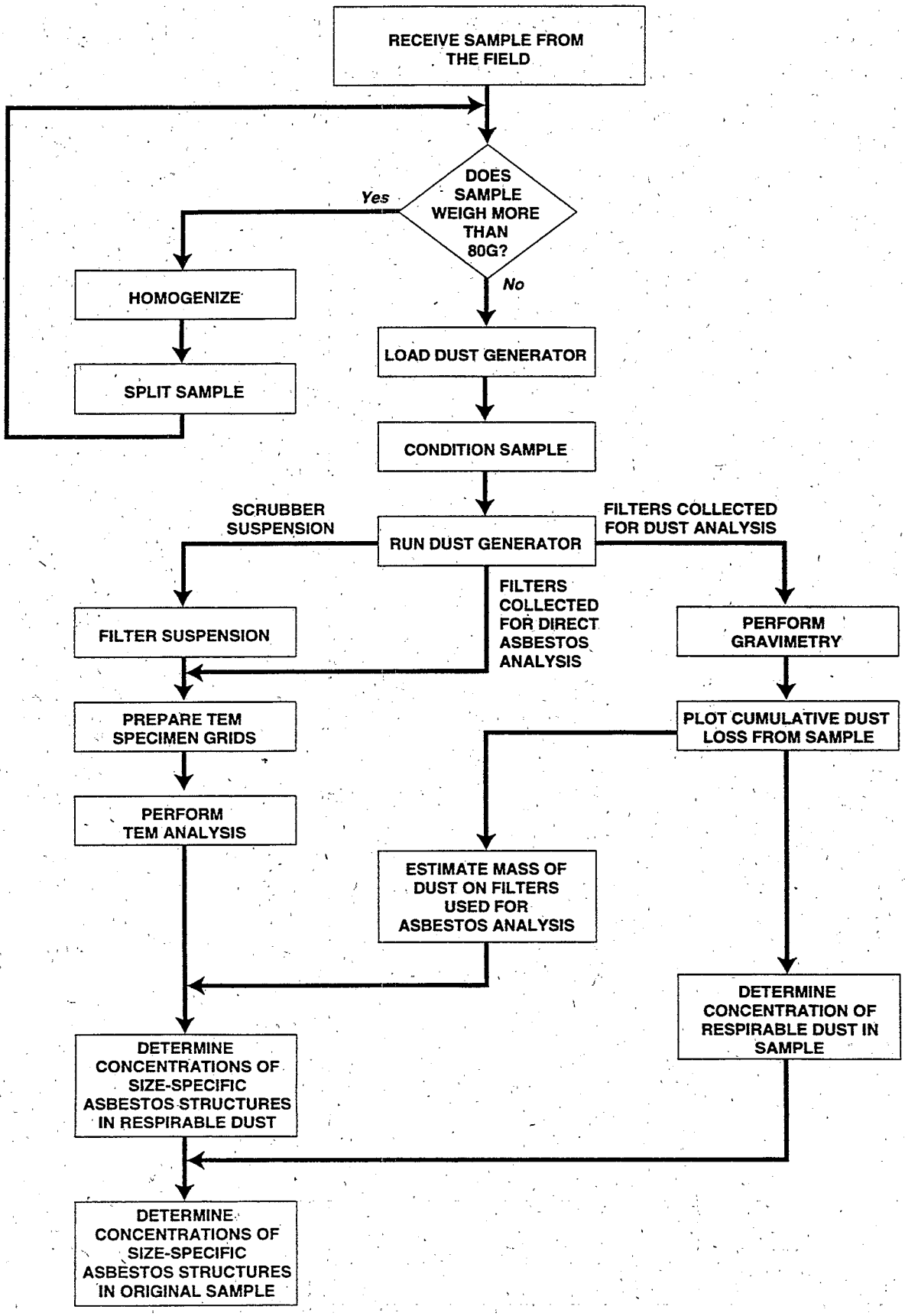


FIGURE 3-2

LABORATORY PREPARATION AND ANALYSIS



While the dust generator is operating, a second set of filters is also collected over the opening of the dust generator that articulates with an isokinetic sampling tube (the center pathway of Figure 3-2). These are collected such that loading is appropriate for specimen grid preparation using a direct transfer technique (Section 9.4.5).

Asbestos structures are also trapped in the suspension of a scrubber during each run of the dust generator (the left side pathway of Figure 3-2). The suspension is then diluted appropriately and filtered to create an additional set of filters from which specimen grids will be prepared for asbestos analysis (Section 9.4.6). However, because asbestos structures derived from this process will have been suspended in the aqueous environment of the scrubber suspension, preparation of grid specimens from filtered scrubber suspension are considered to have been prepared in a manner that is equivalent to an indirect transfer technique.

Next, as indicated in Figure 3-2, TEM specimen grids are prepared using a direct transfer technique from the filters collected either from atop the isokinetic sampling tube of the elutriator or from filtering scrubber suspension (Sections 10.1 and 10.2). Specimen grids are then analyzed using the counting and identification rules of the International Standards Organization (ISO) Method for the determination of asbestos in air using an indirect transfer technique (Chatfield 1993) with the stopping rules modified as indicated in Section 11.1.

Calculations are performed from plots of the cumulative dust loss (Section 11.2) to estimate both the mass of dust co-collected with asbestos on the filters prepared for asbestos analysis and the total mass of respirable dust in the original sample. Dust estimates are then combined with asbestos counts to allow reporting of both the concentration of asbestos structures per unit mass of respirable dust in the sample and the concentration of asbestos structures per unit mass of the original sample (Figure 3-2)[7]. Typically, asbestos concentrations will be reported from this method for a specific size range of asbestos structures of interest.

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[7] When asbestos concentrations are to be reported as a function of the mass of the original sample, the concentration calculated in the laboratory, which represents the concentration of asbestos in the fine fraction of the original sample, must ultimately be adjusted to account for the mass of the coarse fraction of the sample as well (Section 11.4.3).

4.0 DEFINITIONS

Amphibole: a group of rock-forming ferromagnesium silicate minerals, closely related in crystal form and composition, and having the nominal formula:

$$A_{0-1}B_2C_5T_8O_{22}(OH,F,Cl)_2$$

where:

A = K, Na;

B = Fe^{2+}, Mn, Mg, Ca, Na;

C = Al, Cr, Ti, Fe^{3+}, Mg, Fe^{2+};

T = Si, Al, Cr, Fe^{3+}, Ti.

In some varieties of amphibole, these elements can be partially substituted by Li, Pb, or Zn. Amphibole is characterized by a cross-linked double chain of Si-O tetrahedra with a silicon: oxygen ratio of 4:11, by columnar or fibrous prismatic crystals and by good prismatic cleavage in two directions parallel to the crystal faces and intersecting at angles of about 56° and 124° (see Chatfield 1993).

Amphibole Asbestos: amphibole in an asbestiform habit.

Analytical Sensitivity: the calculated asbestos concentration in soil or a bulk matrix, in asbestos structures/g, equivalent to counting of one asbestos structure in the analysis.

Asbestiform: a specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

Asbestos: a term applied to a group of fibrous silicate minerals that readily separate into thin, strong fibers that are flexible, heat resistant and chemically inert.

Asbestos Component: a term applied to any individually identifiable asbestos sub-structure that is part of a larger asbestos structure.

Asbestos Structure: a term applied to any contiguous grouping of asbestos fibers, with or without equant particles.

Aspect Ratio: the ratio of the length to width of a particle.

Blank: a fiber count made on TEM specimen grids prepared from an unused filter (or a filter through which asbestos-free water has been passed), to determine the background measurement. Blanks consist of filter blanks, field blanks and laboratory blanks. Laboratory blanks for this method may include scrubber blanks.

Bundle: a fiber composed of parallel, smaller diameter fibers attached along their lengths (see Chatifield 1993).

Chrysotile: the asbestiform habit of a mineral of the serpentine group that has the nominal composition:

$$Mg_3Si_2O_5(OH)_4$$

In some varieties of chrysotile, the silicon may be partially substituted by Al or less commonly by Fe. The magnesium may be partially substituted by Fe, Ni, Mn or Co. Some varieties contain Na, Cl or both. Chrysotile is a highly fibrous and silky variety and constitutes the most prevalent type of asbestos (see Chatfield 1993).

Cluster: an assembly of randomly oriented fibers (see Chatfield 1993).

Component Count: for any sample, a tally that includes the individually identified components of complex asbestos structures and each single asbestos structure with no identifiable components.

Elutriator: a device in which differential flow through a fluid (gas or liquid) against an opposing force (i.e. gravity) is employed to separate particles by size.

Equant Particle: as used in this document, a non-asbestos particle bound to, or overlapping with, asbestos structures observed on a TEM specimen grid.

Fiber: an elongated particle that has parallel or stepped sides. In this method, a fiber is defined to have an aspect ratio equal to or greater than 5:1 (see Chatfield 1993).

Fibril: a single fiber of asbestos that cannot be further separated longitudinally into smaller components without losing its fibrous properties or appearance.

Fibrous Structure: a contiguous grouping of fibers, with or without equant particles.

Field Blank: a filter cassette that has been taken to the sampling site, opened, and then closed. Such a filter is analyzed to determine the background asbestos structure count for measurement and to document the treatment of the filter from sample collection through analysis.

Filter Blank: an unused filter that is analyzed to determine the background asbestos structure count on the filter matrix.

Friable: as used in this document, capable of being crushed or deformed with the hand with the attendant release of fibers.

Habit: the characteristic crystal form or combination of forms of a mineral, including characteristic irregularities.

Identify: during asbestos analysis, the use of a sequential set of procedures to determine and confirm the mineralogy of a structure.

Isokinetic Sampling: sampling air in such a manner so as not to disturb the direction or velocity of air flow at the point sampled.

Isokinetic Sampling Tube: a tube placed in the air flow of the vertical elutriator portion of the dust generator used in this method, which samples the air at the top of the elutriator isokinetically.

Laboratory Blank: an unused filter that is analyzed along with sample filters to determine the background asbestos structure count in the laboratory.

Matrix:[8] A connected assembly of asbestos fibers with particles of another species (non-asbestos) (see Chatfield 1993).

PCM Equivalent Structure: A structure of aspect ratio greater than or equal to 3:1, longer than 5 μm, and which has a mean diameter between 0.2 μm and 3.0 μm for a part of its length greater than 5 μm. In this method, PCME structures also must contain at least one asbestos component (see Chatfield 1993).

Riffle Splitter: a device composed of a hopper and multiple, uniform, parallel chutes that alternately feed from the hopper to opposing receiving trays.

Scrubber: a device for removing particles from an air stream by passing the air stream through a super-saturated vapor in which the particles serve as nucleation centers for condensation and are thus captured. The resulting droplets (containing the trapped particles) then fall back into a central reservoir of boiling liquid.

Serpentine: a group of common rock-forming minerals having the nominal formula:

$$Mg_3Si_2O_5(OH)_4$$

Serpentine deposits often contain chrysotile asbestos (which is serpentine in an asbestiform habit).

Structure Count: for any sample, a tally of each individually identified asbestos structure regardless of whether the structure contains identifiable components. This is equivalent to a count of the total number of separate asbestos entities encountered on the sample.

Vertical Elutriator: see Elutriator.

Tumbler: a device that is rotated to provide continuous agitation to a bulk material placed inside. In the dust generator employed in this method, air is blown through a tumbler containing sample to carry away the dust generated during agitation by the tumbler.

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[8] When used to describe an asbestos structure. The term is also used in this document to describe a heterogeneous bulk solid.

5.0 SYMBOLS AND ABBREVIATIONS

5.1 SYMBOLS

A_f - the area of a filter from which a specimen grid is prepared (mm^2).

A_{go} - the average area of a specimen grid opening (mm^2).

C_{dust} - the concentration of asbestos structures (of a defined size and type) in the respirable dust from a sample (s/g_{dust}).

C_{mtrx} - the concentration of asbestos structures (of a defined size and type) in the original field matrix that was sampled for analysis using this method.

C_{smpl} - the concentration of asbestos structures (of a defined size and type) in a soil or bulk sample (s/g).

CF - the coarseness adjustment factor representing the ratio of the mass of the fine fraction to the total mass of a matrix that is sampled in the field.

cm - centimeter (10^{-2} meter).

cm^2 - square centimeter.

cm^3 - cubic centimeter.

cm^3/min - cubic centimeter per minute.

d - the density of a particle (g/cm^3).

DF - the dilution factor representing the ratio of the scrubber suspension volume to the aliquot that is filtered to prepare specimen grids.

^oC - degrees centigrade.

^oK - degrees Kelvin.

ΔM_f - the mass of respirable dust collected on a single filter during the interval Δt (g).

ΔM_s - the mass of respirable dust released from the sample during the interval Δt (g).

Δt - a short time interval (no more than 10 minutes).

η - the dynamic viscosity of air (g/cm*s).

eV - electron volt.

F_c - the rate of airflow (i.e. the volumetric flow rate) through the top exit (ME) opening of the elutriator that does not pass through the isokinetic sampling tube (cm^3/s).

F_d - the rate of airflow (i.e. the volumetric flow rate) through the top exit (IST) opening of the elutriator that passes through the isokinetic sampling tube (cm^3/s).

F_s - the rate of airflow (i.e. the volumetric flow rate) through the scrubber (cm^3/s).

ft - foot.

g - gram.

g - the acceleration due to gravity (cm/s^2), when used as a variable in an equation.

g/L - gram per liter.

g/cm^3 - gram per cubic centimeter.

hp - horsepower.

k - the first order rate constant (s^{-1}).

kg - kilogram (10^3 gram).

kV - kilovolt.

in - inch.

L - liter.

L/min - liters per minute.

M_{coarse} - the mass of the coarse fraction of a matrix sampled in the field.

M_f - the cumulative mass of respirable dust collected on filters from the start of a run to time, t (g).

M_{f30} - the cumulative mass of respirable dust collected on filters during an entire 30 rpm run (g).

M_{fine} - the mass of the fine fraction of a matrix sampled in the field.

M_o - the mass of respirable dust in a sample at the start of a run (g).

M_r - the cumulative mass of respirable dust released from a sample from the start of a run to time, t (g).

M_s - the mass of respirable dust remaining in a sample during a run but after time, t (g).

M_{scrbr} - the mass of respirable dust collected in the scrubber during a run (g).

M_{sample} - the mass of a sample introduced into the dust generator (g).

M_{tot} - the total mass of respirable dust estimated to reside in a sample (g).

ml - milliliter (10^{-3} L).

mm - millimeter (10^{-3} meters).

mm^2 - square millimeter.

µg - microgram (10^{-6} grams).

µm - micrometer (10^{-6} meters).

N_{go} - the number of grid openings counted during a scan (#).

N_{goh} - the number of grid openings counted during a high magnification scan (#).

N_{gol} - the number of grid openings counted during a low magnification scan (#).

nm - nanometer (10^{-9} meter).

P_f - the pressure measured at a flowmeter (torr).

P_t - the pressure estimated at an elutriator opening (torr).

%RD - the mass percent of respirable dust in a sample (%).

r - the radius of a particle (cm).

r^2 - the coefficient of determination (also defined as the correlation coefficient squared).

R_f - the flow reading from a flowmeter (cm/s).

S_{ch} - the number of asbestos structures (of a defined size and type) counted during a high magnification scan (#).

S_{cl} - the number of asbestos structures (of a defined size and type) counted during a low magnification scan (#).

S_d - the number of asbestos structures that must be detected during a TEM scan for asbestos to be defined as detected (#).

S_{smpl} - the required analytical sensitivity for this method (defined separately for total and long asbestos structures) (s/g).

s - second.

S/g - structures per gram.

S/g_{dust} - structures per gram of dust.

S/L - structures per liter.

S/mm^2 - structures per square millimeter.

t - time (s).

T_f - the temperature at a flowmeter (^oK).

T_t - the temperature at an exit opening of the elutriator (^oK).

V_{a1} - the volume of the first aliquot collected from the scrubber suspension to be used for further dilution (ml).

V_{a2} - the volume of the final aliquot collected from V_d, which is filtered for the preparation of specimen grids for TEM analysis (ml).

V_d - the volume into which the first aliquot from the scrubber suspension is diluted (ml).

V_l - linear air flow rate (cm/s).

V_s - the volume of scrubber suspension generated from a run (ml).

V_v - the volumetric air flow rate (cm^3/s).

W - watt.

5.2 ABBREVIATIONS

ED - Electron diffraction

EDXA - Energy dispersive X-ray analysis

FWHM - Full width at half maximum

HEPA - High efficiency particle absolute

IST - refers to the opening at the top of the elutriator that is associated with the *isokinetic sampling tube*

MCE - Mixed cellulose ester

ME	-	refers to the *main exit* opening at the top of the elutriator, which is *not* associated with the isokinetic sampling tube
PCM	-	Phase contrast optical microscopy
PCME	-	Phase contrast microscopy equivalent
PLM	-	Polarized light microscopy
RPM	-	Revolutions per minute
SAED	-	Selected area electron diffraction
TEM	-	Transmission electron microscopy
TSP	-	Total suspended particulate
UICC	-	Union Internationale Contre le Cancer

6.0 FACILITIES AND EQUIPMENT

6.1 SAMPLE COLLECTION EQUIPMENT AND CONSUMABLE SUPPLIES

To complete field sampling per this method, the following field equipment is mandatory:

- survey equipment appropriate to the manner in which sample locations are to be defined per the sampling plan;
- appropriate trowels, shovels, augers, or corers for sample collection per the sampling plan;
- (when sampling surface materials) a 12 in square aluminum template with an 8 in square hole in the center;
- a minimum of three 3-gal plastic buckets;
- a brass or steel sieve with 3/8 in. (1 cm) openings;
- a field balance (with a capacity of 40 kg and capable of achieving a precision of ± 10 g);
- a field balance (with a capacity of 2 kg and capable of achieving a precision of ± 0.2 g)[9]
- a riffle splitter with a minimum of 24, 3/4 in. (minimum size) chutes and three sample trays;
- one L plastic sample containers;
- sufficient plastic coolers to store and ship samples at ice temperature;
- equipment for cleaning sampling tools, including:
  - large buckets and tubs;
  - a container of asbestos-free water;
  - garden sprayers;
  - bio-degradable detergent;
  - assorted asbestos-free rags, sponges, etc.;
  - an air compressor with HEPA filter (optional, for drying equipment);
- field logbook and appropriate custody forms and sample labels;
- assorted garbage bags, paper towels, and tape;
- Tyvek suits and protective gloves; and

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[9] If appropriate equipment is available, it is advantageous to use a single field balance to achieve both sets of capacity and precision requirements for field weighing.

- appropriate equipment for respiratory protection.

6.2 LABORATORY FACILITIES

Laboratories wishing to adopt this method must develop and maintain the following facilities:

- a properly ventilated room for bulk sample handling that is entirely isolated from other room(s) in which air samples are handled and asbestos samples are analyzed. All such facilities must be sufficiently well ventilated to allow preparation of blanks that yield background determinations satisfying the requirements of Section 10.6 of the Superfund air method (Chatfield and Berman 1990);

- a glove box or equivalent isolation chamber of sufficient size to house a riffle splitter (or other equipment) required for the homogenization and sub-sampling of samples for this method. The glove box or isolation chamber must provide ample room for handling kg size soil or bulk samples while maintaining background concentrations in the outside room air at levels considered acceptable as defined in Section 10.6 of Chatfield and Berman (1990);

- a dust generator constructed per the specifications provided in Appendix A;

- a TEM operating at an accelerating potential of 80-120 kV, with a resolution better than 1.0 nm and a magnification range of approximately 300 to 100,000. The ability to obtain a direct screen magnification of about 100,000 is necessary for inspection of fiber morphology; this magnification may be obtained by supplementary optical enlargement of the screen image by use of a binocular if it cannot be obtained directly. The TEM shall also be equipped with an energy dispersive X-ray analyzer capable of achieving a resolution better than 175 eV (FWHM) on the MnK_{α} peak. For requirements concerning screen calibration and SAED and ED performance, see Chatfield and Berman (1990); and

- a computer system for recording analytical results. As indicated in the section addressing reporting requirements (see Chapter 13), analytical results are to be provided on computer disk (either 3.5 inch or 5.25 inch in double sided or high density format) in a file format that is compatible with LOTUS™. ASCII files are acceptable.

6.3 THE DUST GENERATOR AND APPURTENANT EQUIPMENT

The dust generator is to be constructed per the design drawings and specifications provided in Appendix A. Appurtenant equipment required to support the dust generator includes:

- a 129 hp DC motor (rated for 0 to 139 rpm) to drive the tumbler;

- two vacuum pumps capable of drawing 20 L/min at minimum load (will be run at 1 to 2 L/min);

- two variable area flowmeters capable of reading volumetric airflow velocities up to 1500 ml/min and one variable area flowmeter capable of reading airflow up to 250 ml/min;

- a heating mantle and variable voltage transformer suitable for maintaining water at a boil in a 1 L round bottom flask; and

- an immersion pump and cooler (or equivalent system) of sufficient capacity to provide 0^{o} C water at a rate of 1 to 2 L/min.

6.4 SPECIMEN PREPARATION EQUIPMENT

As defined in the ISO Method for the determination of asbestos in air using an indirect transfer technique (Chatfield 1993).

6.5 OTHER LABORATORY EQUIPMENT

As defined in the ISO Method for the determination of asbestos in air using an indirect transfer technique (Chatfield 1993).

6.6 CONSUMABLE/REUSABLE LABORATORY SUPPLIES

For each run of a sample using the dust generator:

- a lot of fifty MCE filters (0.45 µm pore size, 25 mm) that exhibit no more than 10 s/mm^2 asbestos as background[10]; and

- forty plastic petri dishes for storing 25 mm filters.

Also, other items as defined in the ISO Method for the determination of asbestos in air using an indirect transfer technique (Chatfield 1993)[11].

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[10] This value is selected to assure that detection of a single structure in 4 grid openings is more likely than not to constitute asbestos from a sample.

[11] This includes a supply of MCE filters (0.22 µm pore size) for filtering scrubber suspension.

7.0 REAGENTS

To support use of the dust generator, the following reagents are required:

- asbestos-free water (a regular supply of freshly distilled, filtered water must be available);
- potassium carbonate dihydrate (analytical grade)[12]; and
- sodium hexametaphosphate (analytical grade).

Also, reagents required to support asbestos analysis are defined in the ISO Method for the determination of asbestos in air using an indirect transfer technique (Chatfield 1993).

WARNING - USE ALL REAGENTS IN ACCORDANCE WITH THE APPROPRIATE HEALTH AND SAFETY REGULATIONS.

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[12] This salt is required for loading into the constant humidity chamber and the desiccators to be used for conditioning filters under the recommended default conditions for running the dust generator. A supply of alternate salt (analytical grade) may be employed for studies in which dust generation is to be performed at a relative humidity other than the default recommendation (see Section 9.3.2).

8.0 SOIL OR BULK SAMPLE COLLECTION

Sample collection procedures adopted for this method are flexible to allow adequate sampling of a broad variety of matrices. The method also incorporates several field preparation steps that are designed to preserve sample representativeness while reducing the mass of the samples sent to a laboratory for analysis. Controlling the mass of the samples sent to a laboratory from the field is a cost saving measure (see Section 8.2).

WARNING:

MOST OF THE SAMPLE COLLECTION PROCEDURES AND FIELD PREPARATION PROCEDURES DISCUSSED IN THIS DOCUMENT ARE INHERENTLY DUSTY OPERATIONS. THEREFORE, WHEN HANDLING SOILS OR BULK MATERIALS THAT ARE KNOWN TO CONTAIN OR POTENTIALLY CONTAIN ASBESTOS, IT IS IMPERATIVE THAT PROPER RESPIRATORY PROTECTION BE WORN WHILE CONDUCTING THESE PROCEDURES.

8.1 SAMPLE COLLECTION

Any variety of commercially available field sampling equipment (trowels, shovels, augers, corers, etc.) may be used to collect samples for this method. The equipment and procedure(s) selected should be based on the nature of the material being sampled and the depths over which samples are to be collected. Two common examples are presented below. Whatever equipment and procedures are chosen, however, shall be applied consistently and invariably at each sampling location[13].

Whatever technique is chosen, the *minimum* size sample that shall be collected at each sampling location shall be 1 kg. Larger samples may be required, however, if particularly large (i.e. larger than a 4 or 5 cm in diameter) rocks or debris are present in the material being sampled. To assure representativeness, the largest component sampled should occupy no more than a few percent of the volume of the sample collected (Section 2.3). If samples are to be composited (Section 8.2), they shall be of similar mass (i.e. differences in mass between composited samples shall be no larger than 10% of the mass of the smallest sample).

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[13] Locations from which soil or bulk samples are to be collected shall be selected formally as part of a comprehensive strategy that is designed to provide a representative (unbiased) set of measurements for characterizing the releases of asbestos from the entire source matrix of interest. Procedures for designing such a strategy are beyond the scope of this document but are available elsewhere (see, for example, Berman and Chesson undated).

It is particularly critical, when collecting samples for soil or bulk asbestos analysis, to minimize field decisions that might alter the locations for sample collection that have been selected as part of a formal strategy. Such locations should be representative of the variation of *all* characteristics of the sampled matrix that might affect asbestos release. It is inappropriate, for example, to adjust the location of a sample just because a large rock happens to be located within the footprint over which the sample is supposed to be collected; the presence of that rock as part of that sample helps to represent the fraction of the surface of the sampled matrix from which asbestos release *cannot* occur.

When used to represent the central characteristics of a large matrix, random or systematic sampling schemes depend on faithfully preserving the consequences associated with the choice of *each* sampling location. In general, therefore, it is not appropriate to alter the selected locations even if collection of a sample at a specific location is impossible. Rather than altering such a location, any difficulties or interference that may hinder sample collection at a defined location shall be noted in detail in the field log book.

All sampling equipment shall be washed thoroughly with water and detergent between collection of each sample. Sampling equipment shall then be rinsed thoroughly with filtered, distilled water and allowed to air dry. Forced air may be used to expedite drying. If forced air is to be used to facilitate drying, however, such air must be passed through a HEPA filter to prevent delivery of any potential contamination.

Record the identification number, the date, time, and method of collection for each sample in a field notebook. Record the locations from which each sample is collected in the field notebook. Note in the logbook any changes between the sampling locations proposed in the sampling strategy and the actual locations sampled. As indicated previously, such changes are to be avoided to the extent possible. If changes are absolutely necessary, clearly document the rationale behind each change.

Supplement written documentation with photographs of each sampling location. This is particularly important if the sampling locations are not laid out on a formal, documented sampling grid that is tied to a permanent field marker.

8.1.1 Sampling to Derive Estimates of Asbestos Concentrations in a Road Surface

To illustrate the important features of sampling for this method, assume that sampling is to be performed to determine the concentration of releasable asbestos in the material of a serpentine-covered road. In this case, it is assumed that measured asbestos concentrations are to be related to current emissions so that it is only the actual surface layer of the road that is of interest.

Sampling a road surface shall be conducted using a metal template to define the bounds of each sample and a trowel (or other digging device) to remove material within the template to a uniform depth of 0.5 in. (approximately 1.5 cm). To assure that the samples collected exceed 1 kg in mass (assuming a density for unconsolidated serpentine of 2.2 g/cm^3), the sample volume should be a minimum of 450 cm^3 (28 in^3). Assuming, as indicated above, that each sample will be excavated to a uniform depth of 0.5 in., an 8 in. square hole in a 12 in. aluminum template works well.

At *each* selected sampling location, center the template on the defined location and press it firmly against the ground surface. Carefully excavate the material within the template to a uniform depth of 0.5 in. and place the material in a clean, pre-weighed bucket (see Section 8.2.1)[14]. Transport the sample to a central location on the site where field preparation (Section 8.2) will be performed.

8.1.2 Sampling a Mine Tailings Pile to Derive Estimates of Asbestos Concentrations Within the Pile

As a second illustration of sampling to support this method, assume that sampling is to be performed to determine the concentration of releasable asbestos in the material of a mine

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[14] Depending on conditions encountered, it may be necessary to remove surface debris (such as leaves or foreign dust) from an area prior to sampling. The determination as to whether or to what extent surface debris needs to be removed prior to sampling should be based on careful inspection of the sampling location and review of the motivation for sampling. Surface debris should generally *not* be removed unless such material is clearly distinguished visually from the matrix material to be sampled.

tailings pile. Assume further that the goal is to predict long-term emissions from the pile based on the asbestos concentrations measured. In this case, it is the concentration of asbestos within the volume of the entire pile that is of interest.

For the assumed tailings pile, conduct sampling using a hand auger (if the pile is no thicker than a few feet) or a power auger or other drilling equipment (if the pile is significantly thicker than a few feet). A coring cylinder that is 12 in. long and 2 in. in diameter (assuming an average density for the pile material of 2.2 g/cm^3) yields a sample of approximately 1.4 kg. Such a cylinder shall be collected from each location (defined in 3-dimensions: longitude, latitude, and depth) that is selected for sampling within the pile.

Cores shall be driven until their mid-point overlies the proposed sampling location. Collect the core carefully from each selected location, being sure that the entire core is extracted from the pile. Transport the sample to a central location on the site where field preparation (Section 8.2) will be performed.

8.2 FIELD PREPARATION

Although the following activities might conceivably be conducted in the laboratory (following collection and shipment of kg size samples), to minimize the mass of samples sent to the laboratory and thereby reduce costs, the following field preparation activities are incorporated into this method. For special cases, assuming that the laboratory of choice has access to the required equipment in the required protective enclosures, these activities may be conducted in the laboratory rather than in the field.

Per the instructions in the following sections, samples that are collected as defined in Section 8.1 are to be:

- weighed;
- sieved to separate a coarse and fine fraction;
- the coarse and fine fractions are to be weighed;
- the fine fraction is to be homogenized and split; and
- each of the two final sub-sample splits of the fine fraction are to be weighed, packaged, and shipped to the laboratory (as paired duplicates).

8.2.1 Weighing

As indicated in Section 8.1, samples shall be transported in clean, pre-weighed buckets from the locations at which they were collected to a central location for field preparation. The first step of the process shall be to weigh each sample.

Kg size samples are to be weighed using a field scale capable of reading mass with a minimum precision of ± 10 g. If necessary, wipe the outside surface of each sample bucket with a clean, dry (asbestos free) cloth before placing it on the scale (Figure 8-1). Record the mass measured for each sample (along with its identification number) in a field notebook and subtract the tare weight of the bucket to derive the net weight of the sample.

FIGURE 8-1

WEIGHING A SOIL SAMPLE ON A FIELD SCALE

Following size reduction (Section 8.2.2), both the coarse and fine fractions of each sample are to be weighed again. If necessary, wipe the outside surfaces of the buckets containing each size fraction with a clean, dry (asbestos free) cloth before placing it on the scale. Record the measured weight for each fraction in the field notebook under the appropriate sample identification number. Subtract the tare weight of each bucket holding, respectively, the coarse and fine fractions from the sample and record the net weights of each in the field notebook.

Following homogenization and splitting (Section 8.2.3), sub-samples that are to be sent to the laboratory are to be weighed yet again. In this case, sample weights are expected to range between approximately 50 and 80 g (see Section 9.1) and will need to be measured on a scale that can achieve a precision of ± 0.2 g. Depending on the types of equipment available to the sampling team, this may or may not be the same scale that is used for weighing the initial (heavier) samples. When sending 50 to 80 g splits to the laboratory, both halves of the final sample split shall be sent to the laboratory as a duplicate pair.

Wipe the outside surface of each sample container with a clean, dry (asbestos free) cloth before placing it on the scale. Record the weight, identification number, and pedigree of each sample split of each duplicate pair sent to the laboratory in the field notebook (Figure 8-2). Subtract the tare weight of each sample container from each sample and record the net weight of each sample in the field notebook next to the appropriate sample identifier. Package and send each sample to the laboratory as described in Section 8.4.

8.2.2 Size Reduction

After collection and the initial weighing (described above), pass each sample through a clean, wire-mesh sieve with 1 cm (3/8th in) openings. Reduce all clods and soft aggregates by hand and force all reducible material through the sieve into a clean, pre-weighed bucket (Figure 8-3). Stones and debris retained by the sieve that cannot be hand crushed shall be placed in a separate pre-weighed bucket. Once separated, the coarse and fine fractions from each sample are to be weighed separately (Section 8.2.1).

Clean the sieve with detergent and rinse with filtered, distilled water between samples. Dry the sieve with an asbestos free cloth or with appropriately filtered, forced air before each use.

8.2.3 Sample Homogenization and Splitting

The fine fraction of samples collected for this method may be homogenized and split by either of two procedures.

FIGURE 8-2

WEIGHING A SAMPLE SPLIT ON A FIELD SCALE

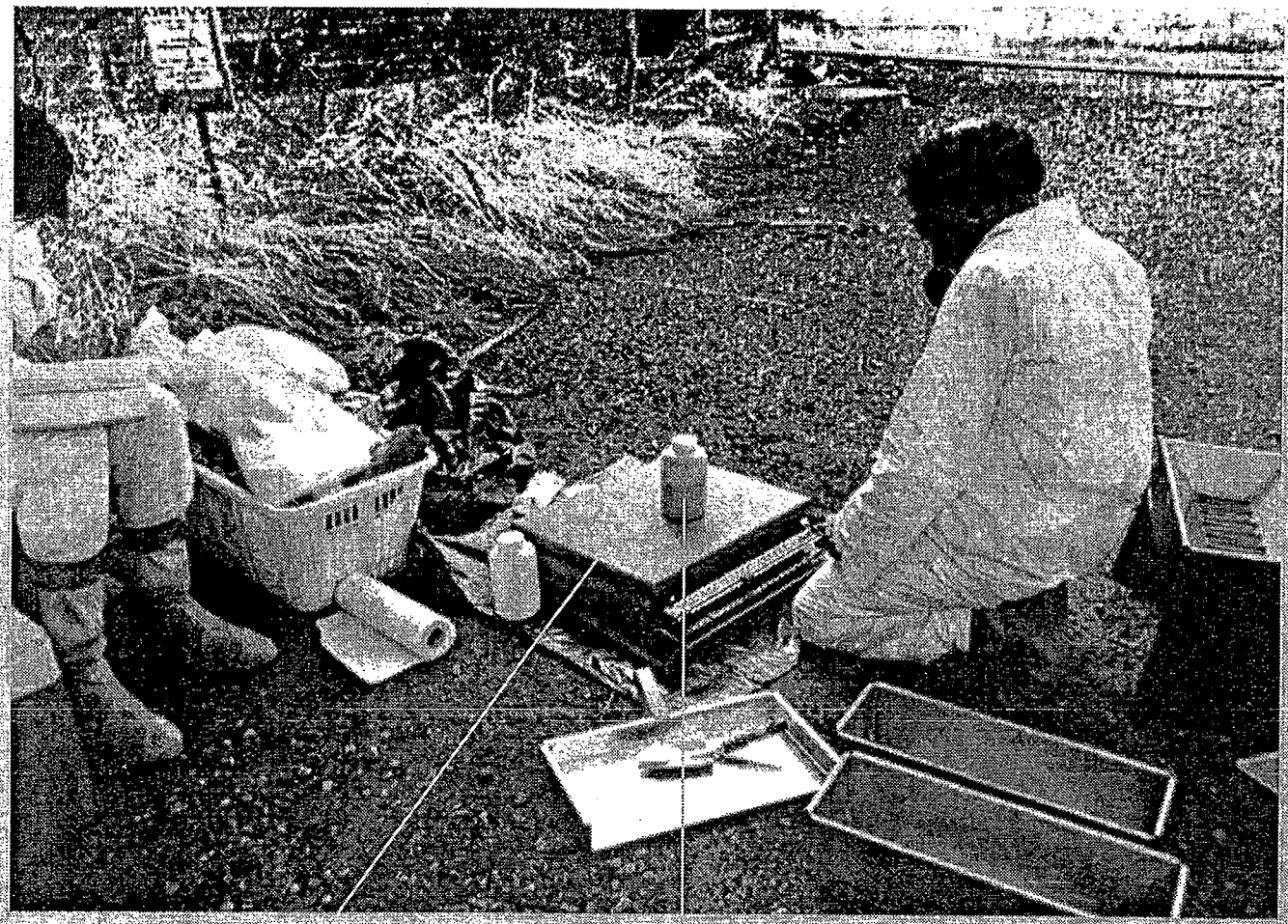

FIGURE 8-3

SIEVING A SOIL SAMPLE

Sieve

Bucket for
Fine Fraction

Option 1: use of a riffle splitter[15]. Set a clean, dry riffle splitter with 3/4 to 1 in. chutes (Figure 8-4) on its stand on flat ground and place two receiving trays under the splitter so that they will each catch material that falls through one of the two sets of chutes (Figure 8-5). Place the sample to be homogenized in a third splitter tray (which must be clean and dry). Shake the splitter tray gently, until the sample is evenly distributed within the entire tray.

Place the long lip of the tray containing the sample against the inside of the long lip of the splitter hopper and slowly rotate the tray along an access defined by its lip so that the sample slowly empties into the splitter and slides down the near wall of the hopper to the chutes (Figure 8-5). Continue to rotate the tray until it lies entirely inverted over the top of the hopper of the splitter.

Tap the tray vigorously several times to free any remaining material and remove the emptied tray from the splitter. Tap the splitter vigorously several times to facilitate the flow of all material through the chutes into the receiving trays. If necessary, any sample remaining along any of the soldered corners and nooks of the splitter may be freed with a clean, coarse nylon brush. When brushing is completed, tap the brush vigorously against the splitter wall to free any material clinging to the brush[16]. Remove the two receiving trays (each containing half of the sample) from the splitter.

What is to be done next depends on whether the goal is to homogenize the sample or to split the sample. If the sample is in the process of being homogenized, combine the half of the sample from each receiving tray back into the third tray from the splitter. Be sure to tap each tray vigorously to assure quantitative transfer of the sample material. Replace the two *empty* receiving trays under the splitter and repeat the process of splitting the sample (in the manner described above). The sample should be subjected to a minimum of five cycles to assure adequate homogenization.

If the goal is to split (rather than homogenize) the sample, pour the material in one of the receiving trays from the splitter into a spare bucket and tap the tray vigorously to assure quantitative transfer. The remaining tray that still contains sample material now becomes the new sample tray and the original sample tray (now empty), along with the just emptied receiving tray, should be placed under the splitter as the new receiving trays.

Repeat the process of dispersing the remaining sample material (containing half the mass of the original sample) by shaking the sample tray so that it is uniformly distributed. Repeat the procedure described above for splitting the sample, discarding the material in one of the two receiving trays each time, until the mass of the material in each receiving tray at the end of one cycle falls in the range of 50 to 80 g. At that point, carefully transfer the material from

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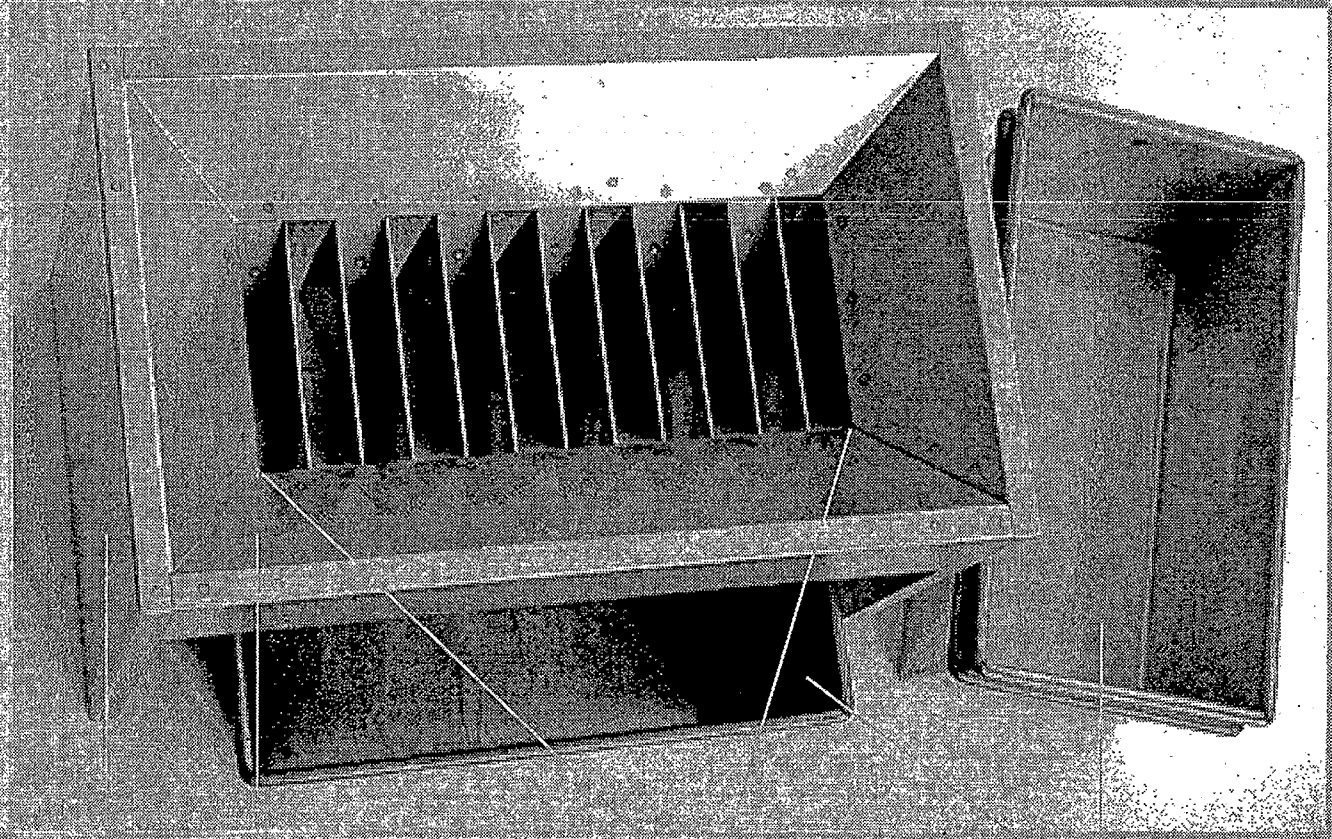
[15] Results obtained as part of the pilot study for this method (Berman and Kolk 1994) suggest that some respirable dust may be lost each time a sample is passed through a riffle splitter. If the process is conducted carefully, however, such loss may be kept sufficiently small so that the multiple passes required to homogenize and split a sample properly will not significantly alter the estimated concentration of dust and asbestos derived using this method; losses should be less than an absolute maximum of 10 to 15% of the total respirable dust in the sample after as many as 10 passes. Because the estimate of the magnitude of loss was necessarily based on measurements of samples that had to be suspended in water, however, actual losses for most cases are expected to be much smaller.

One important consideration: avoid using the splitter in the field on windy days (i.e. when wind velocities exceed approximately 5 mph) unless an effective wind screen can be devised.

[16] The brush will have to be washed, rinsed, and dried thoroughly before use on another sample.

FIGURE 8-4

A RIFFLE SPLITTER



Stand Hopper Chutes Sample Trays

FIGURE 8-5

USING A RIFFLE SPLITTER TO HOMOGENIZE/SPLIT SAMPLES

Sample Tray Receiving Trays Splitter Hopper Splitter Stand

each tray into a clean, pre-weighed sample bottle (Figure 8-6) to be weighed (Section 8.2.1) and packaged for shipment to the laboratory (Section 8.4). Be sure that samples are transferred quantitatively from each tray.

NOTE

***NEVER* MAKE A PARTIAL TRANSFER OF MATERIAL FROM A SPLITTER TRAY TO A RECEIVING CONTAINER. THIS WOULD DEFEAT THE PURPOSE FOR HOMOGENIZATION AND SPLITTING BECAUSE IT IS IMPOSSIBLE TO ASSURE THAT ALL OF THE SIZE COMPONENTS OF THE SAMPLE ARE TRANSFERRED PROPORTIONALLY UNDER SUCH CIRCUMSTANCES.**

Clean the body of the splitter, all trays, and any appurtenant equipment (such as a nylon brush) between samples (but *not* between splits of the same sample) with a detergent wash followed by thorough rinsing with distilled, filtered water. Be sure that the splitter, trays, and appurtenant equipment are completely dry before use. These may be dried with forced air that is properly filtered to be free of asbestos.

Option 2: Use of a mixer with coning and quartering. Samples may also be homogenized for this method using any of various sealed, rotating mixers (tumblers). The mixers should contain internal baffles to promote mixing. Such mixers must be sufficiently large to accommodate the largest sample to be homogenized[17] with adequate room to spare so that tumbling is facilitated. The mixers must be sealable to prevent the loss of fines during mixing.

Place the fine fraction of the sample to be homogenized in a clean, dry mixer. Seal the mixer. Tumble the mixer at the manufacturers recommended speed for an amount of time recommended by the manufacturer to assure adequate homogenization. Stop the mixer and allow ample time (approximately 15 minutes) for the fines to settle. Disconnect the mixing container from the rest of the mixer. Open the mixer.

Under this option, a procedure termed coning and quartering is used to split a homogenized sample. Lay out a clean, aluminum plate on a flat surface. Hold the mixer immediately over the center of the plate and rotate the mixer around an axis represented by the lip on one side of its mouth so that the sample material *slowly* pours onto the metal plate forming a symmetrical cone (Figure 8-7). Keep the point at which the poured material impacts the cone at the same spot and slowly raise the mixer as the pouring continues to keep the distance between the mixer lip and the top of the cone approximately constant. When the mixer is fully inverted, tap it vigorously to complete the quantitative transfer.

To halve the cone, hold a second (clean, dry) aluminum plate directly over the apex (top center) of the cone at an angle that is perpendicular to the aluminum plate on which the cone lies. Slowly lower the second plate so that it splits the cone precisely in half (Figure 8-8a). While holding the two plates steady, push one half of the cone off of the original plate and away from the rest of the sample (Figure 8-8b). Brush the area from which this material is

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[17] If samples are to be composited as described in Section 8.3, the mixer may have to be capable of handling samples that range up 40 kg in size.

FIGURE 8-6

LOADING A SAMPLE SPLIT INTO A SAMPLE BOTTLE

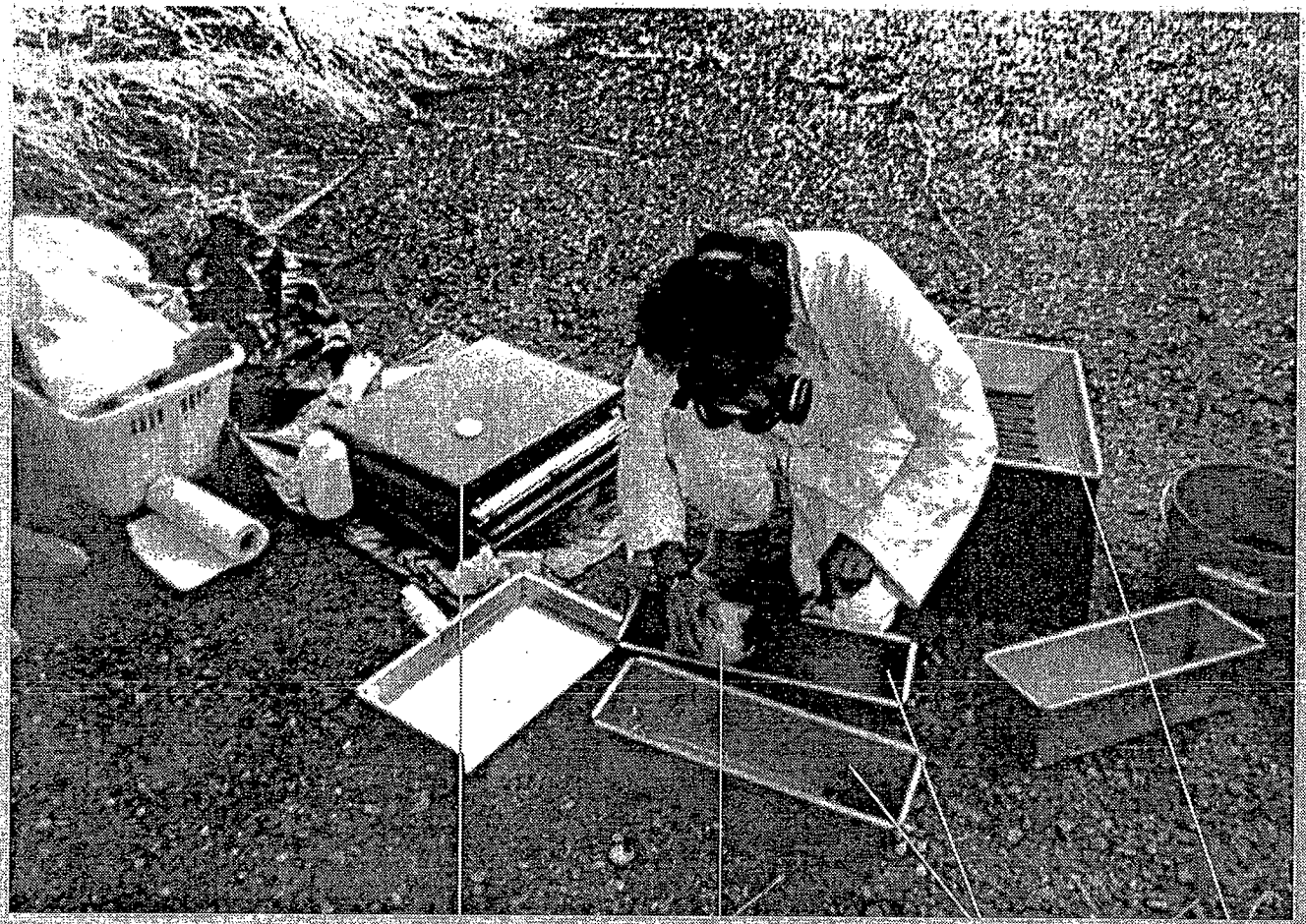


FIGURE 8-7

TRANSFERING SAMPLE FROM A MIXER TO A PLATE FOR CONING AND QUARTERING

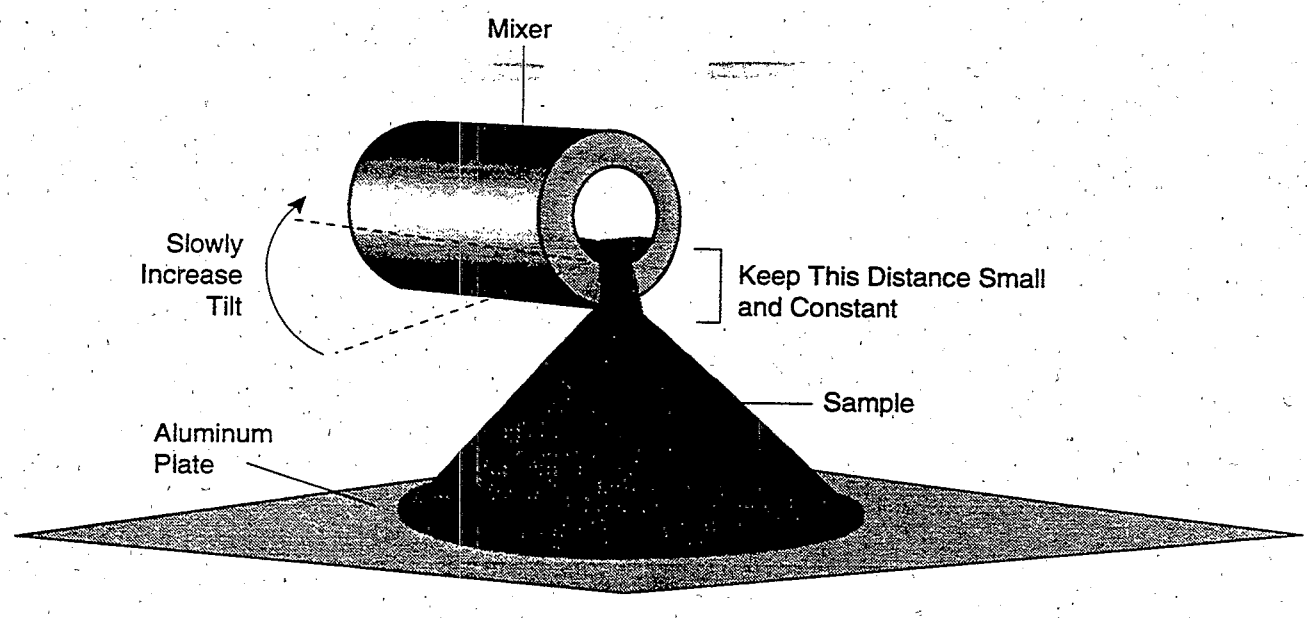
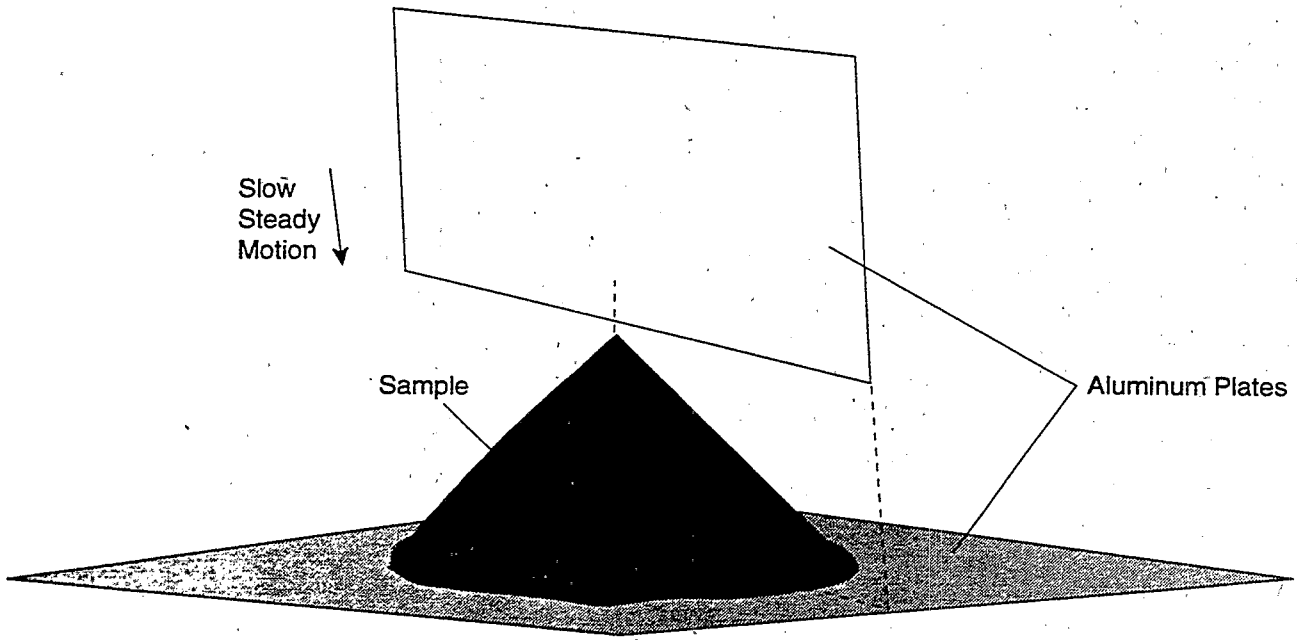
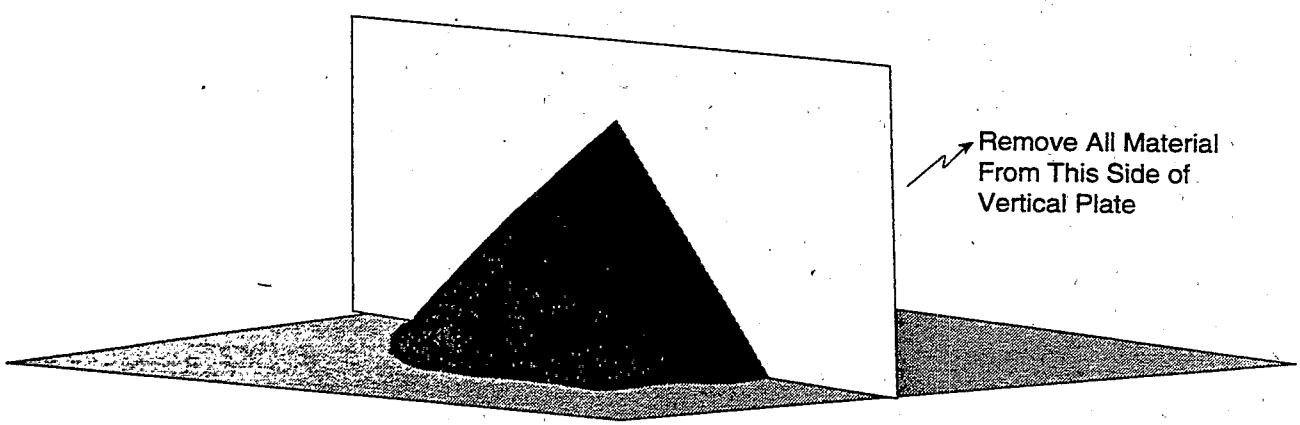


FIGURE 8-8

CONING AND QUARTERING



A. POSITION OF ALUMINUM PLATES IN PREPRATION FOR HALVING SAMPLE



B. POSITION OF ALUMINUM PLATES AFTER HALVING SAMPLE

removed to complete a quantitative transfer, leaving only clean metal. Once half of the sample material has been removed, withdraw the vertical aluminum plate slowly by pulling it upward vertically.

To complete quartering of the cone, rotate the vertical aluminum plate above the cone 90° on a vertical axis. Lower the plate slowly once again so that it splits the remaining portion of the sample cone evenly into two new halves. Once again, quantitatively remove half of the sample material (i.e. remove all of the material from one side of the vertical plate). This process may be repeated by quantitatively transferring the remaining sample material into a clean bucket and pouring the sample onto the clean aluminum plate to form a new cone.

Repeat the coning and quartering process until the remaining quarters (or halves) of the sample at the end of one cycle falls in the range of 50 to 80 g. At that point, carefully transfer the material from each quarter (or half) into a clean, pre-weighed sample bottle to be weighed (Section 8.2.1) and packaged for shipment to the laboratory (Section 8.4).

NOTE

NEVER MAKE A PARTIAL TRANSFER OF MATERIAL FROM ANY PORTION OF THE CONE THAT DOES NOT INCLUDE A WEDGED-SHAPE SLICE THROUGH THE CENTER OF THE CONE OVER ITS ENTIRE THICKNESS (DEPTH). THIS WOULD DEFEAT THE PURPOSE FOR FORMAL CONING AND QUARTERING BECAUSE IT IS IMPOSSIBLE TO ASSURE THAT ALL OF THE SIZE COMPONENTS, WHICH WILL *NOT* BE HOMOGENEOUSLY DISTRIBUTED VERTICALLY THROUGHOUT THE CONE OF THE SAMPLE, ARE TRANSFERRED PROPORTIONALLY.

8.3 COMPOSITING SAMPLES (OPTIONAL)

In many cases, there may be interest in limiting the number of analyses required to characterize a matrix that serves as a potential source *without* sacrificing representativeness. One procedure that may be employed for this purpose is to composite samples in the field. Note however, while compositing can reduce the cost of analysis by reducing the number of samples requiring analysis, also lost is information concerning the spatial variability of the sampled matrix. Therefore, if such information is desired for any particular reason, compositing is not recommended.

Only minor adjustments to the field preparation procedures described above are required to incorporate compositing into this method. First, during planning, group the samples to be collected in the field into sets that are to be composited. For example, there may be a desire to combine all samples from the eastern part of a road into a composite representing the east end of the road. Similarly, samples from the west end might be combined into a west end composite. Alternately, all samples from the road may be combined into a single composite, representing the road as a whole. As another alternative, the composite road sample might also be split into duplicate pairs to allow determination the variability contributed by sample preparation and analysis. Such decisions shall all be determined during planning.

NOTE

It is expected that the same set of locations will be selected for sample collection whether or not compositing is employed; compositing only changes the number of analyses required. This is because, the same number of samples collected from a set of locations selected using the same formal procedures are still required to adequately characterize the sampled matrix, whether or not samples are composited prior to analysis.

When brought to the central location where field preparation is conducted, after the initial weighing, the samples collected from a set that is to be composited can be combined in a common bucket. Modify the procedures described in Sections 8.2.1 and 8.2.2 as follows:

- transport each sample (of a set to be composited) from the point of collection to a central location in a clean bucket;

- weigh the sample and record the weight along with the appropriate sample identifier. Subtract the tare weight of the bucket and record the net weight of the sample;

- sieve the sample and collect the fine fraction from each sample in the set to be composited in a common bucket. Weigh the bucket containing the fines following the addition of the contributions of each sample and subtract the previous weight of the bucket to determine the net weight contributed by each sample. Record the weight with the proper identifier in the field notebook; and

- transfer the coarse fraction from each sample in the set to be composited to a common bucket. Weigh the bucket containing the coarse fraction following the addition of the contributions from each sample and subtract the previous weight of the bucket to determine the net weight contributed by each sample. Record the weight of the coarse fraction with the proper sample identifier in the field notebook.

Once all the samples of the set to be composited have been collected and added, the combined fine fraction from all of the samples (which resides in a common bucket) shall be given a separate identifier representing the intended composite. Record the new identifier in the field notebook. This material can now be treated as a single, composite sample for all remaining steps of field preparation and sample handling, packaging, and shipment to the laboratory. Thus, homogenize and split the sample as described in Section 8.2.3 and package and ship the sample to the laboratory as described in Section 8.4. Record the appropriate weights of the samples to be shipped as described in Section 8.2.1.

8.4 SAMPLE HANDLING AND SHIPMENT

Once their weights and identifiers are recorded, the samples to be shipped to the laboratory must be sealed and labeled. Fill out and apply appropriate labels to each sample bottle. Record the date and time that each sample was created on both the label and the field notebook and be sure that the identification numbers on the label and field notebook match.

Fill out the appropriate chain of custody forms and seal each sample cap with a breakaway label. As indicated in Section 12.2, be sure to complete the field activities report and to include this report in the package with samples sent to the laboratory.

Wipe each sample to be shipped to the laboratory with a clean, asbestos-free cloth and place it in a cooler. Ship samples to the laboratory in a cooler with ice to limit biological growth during shipment. Sufficient ice must be provided to assure that samples remain cold until received and processed by the laboratory.

NOTE

When matrices that are sampled contain a significant fraction of coarse material (i.e. more than 10% by mass), the final determination of the concentration of asbestos in that matrix must be adjusted to account for the fraction of coarse material. This requires determining the ratio of the mass of *fine* material in the sampled matrix to the total mass of material in the sampled matrix to generate a "coarseness adjustment factor". The concentration of asbestos determined for samples sent to the laboratory must then be multiplied by this coarseness adjustment factor to determine the concentration of asbestos in the sampled matrix.

Equations for deriving and using the coarseness adjustment factor are provided in Section 11.4.3. The weights of the coarse and fine fractions of each sample are to be included (along with the appropriate sample identifiers) as part of the field activities report that is to be shipped with the sample to the laboratory (Section 12.2). This assures that individuals responsible for estimating the concentration of asbestos for the project have access to the required field information.

9.0 SAMPLE PREPARATION BY DUST GENERATION

The primary purpose for sample preparation by dust generation that is described in this section is to generate dust-laden filters that can be suitably prepared for analysis by an appropriate method for the determination of asbestos in air[18]. The rate of generation of total respirable dust is also monitored and is used both to estimate the total mass of respirable dust in the original sample and to tie asbestos structure concentrations determined from filters to the mass of the original sample. Such information might also be used in some studies to characterize the releasibility of asbestos (or total dust) from particular sample types.

A detailed description of the apparatus employed for dust generation and its theory of operation is provided in Appendix A. Specifications and construction drawings are also provided.

9.1 SAMPLE RECEIVING AND STORAGE

All samples received from the field are to be wiped clean with a damp cloth prior to storage or other handling. Samples shall be stored at ice temperature (to minimize biological growth) until sample preparation is initiated. To minimize complications from biological agents, once initiated, sample preparation shall be completed expeditiously. In any case, sample preparation shall be completed within 48 hours.

Samples to be prepared using the dust generator are to be inspected for the presence of free water. If a sample contains free water or if the sample appears visibly moist, it shall be dried at low temperature. If time permits, place the sample in an open, shallow container and store it for several days in a desiccator containing *moist* potassium carbonate dihydrate or another salt that corresponds to the salt selected for humidity control (see Section 9.3.2). The type of salt placed in the desiccator is chosen deliberately; rather than to dry the sample completely, the goal is to bring the moisture content of the sample into equilibrium with conditions that will prevail in the dust generator.

If sufficient time is not available to dry the sample in a desiccator, the sample may be oven dried. Dry the sample in an open, shallow container in an oven that is maintained at a temperature *below* 60° C until the sample comes to constant weight. Note that oven-dried samples may require additional time for conditioning (Section 9.4.2) because the moisture content of the sample will need to be increased to bring it into equilibrium with conditions prevailing in the dust generator.

Once dry, samples smaller than 80 g can be loaded directly into the tumbler of the dust generator (Section 9.4.1). Larger samples must be homogenized and split, as described in Section 9.2, prior to being placed in the tumbler of the dust generator.

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[18] The ISO method for the determination of asbestos in air (using either an indirect or a direct filter preparation technique -- Chatfield 1993) is the default method recommended for use in tandem with this method.

9.2 SAMPLE HOMOGENIZATION AND SPLITTING IN THE LABORATORY

Samples received from the field that are larger than approximately 80 g must be dried, as described above, and then homogenized and split as described in this section.

WARNING:

BECAUSE ASBESTOS CONTAINING DUSTS MAY BE GENERATED FROM THE HANDLING AND PREPARATION OF BULK SAMPLES, ALL OF THE FOLLOWING PREPARATION STEPS SHALL BE PERFORMED IN A PROTECTIVE ENCLOSURE (I.E. A HEPA FILTERED GLOVE BOX OR AN APPROVED FUME HOOD THAT IS DESIGNED TO MINIMIZE EXPOSURE TO LABORATORY PERSONNEL[19]). IT IS ALSO NECESSARY THAT ALL HANDLING OF BULK SAMPLES BE CONDUCTED IN A SEPARATE ROOM THAT IS PHYSICALLY ISOLATED FROM THE ROOM(S) IN WHICH AIR SAMPLES ARE HANDLED AND ASBESTOS ANALYSIS IS PERFORMED.

As with field homogenization and splitting (Section 8.2.3), either of two options may be selected for homogenization and splitting in the laboratory. When performed in the laboratory, however, such equipment must fit within an appropriately designed, protective enclosure, which is why field preparation may be cost-effective.

Homogenize large samples in precisely the same manner as described in Section 8.2.3. Once samples are homogenized, split samples in precisely the same manner as described in Section 8.2.3. Continue splitting until a paired set of samples are produced that each contain between 50 and 80 g of material. Record in a laboratory notebook the final weights and identification numbers of the samples homogenized and split.

9.3 DUST GENERATOR SETUP

Prior to using the dust generator, a supply of at least 35 MCE filters must be conditioned and stored for use, the constant humidity chamber must be loaded with the appropriate solution, the scrubber must be primed, and air flow within the dust generator must be calibrated and adjusted.

9.3.1 Conditioning a Stock of Filters

A stock of at least 35 filters (0.45 µm pore size, 25 mm diameter), all from the same filter lot, must be conditioned in a desiccator overnight to bring them into equilibrium with the relative humidity at which they will be used during a run. Place the 35 MCE filters in a desiccator containing *moist* salt of the same variety as that selected to fill the pans in the humidity control chamber of the dust generator (Section 9.3.2). For most applications, this will be potassium carbonate dihydrate (see Appendix A).

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[19] The work should be performed in a Class II biohazard hood as per the specifications of Standard #49 of the National Sanitation Foundation.

After storing the filters overnight in the desiccator, pre-weigh each filter to a minimum precision of ± 0.0002 g[20]. Each filter shall then be placed in a separate, covered Petri dish with its weight marked on the top of the container. The lids shall also be numbered sequentially and the filters shall all be used during the run in the order numbered.

9.3.2 Initiating Humidity Control

Use asbestos-free (filtered, distilled) water to make a 2 L solution of saturated salt. As indicated previously, for most applications, use potassium carbonate dihydrate (to achieve a relative humidity of 43%), but other salts may be used for specific applications (see Appendix A)[21].

Prepare the solutions by placing 1000 g of the anhydrous salt into a one L container and adding distilled water to fill the container. The container should be capped and additional water and salt added as necessary the next day. No water or salt should be added within a day of using the mixture since some time is needed to saturate the salt solution and form a hydrate when adding anhydrous potassium carbonate. However, sufficient salt shall have been added previously to assure that sufficient undissolved potassium carbonate dihydrate has precipitated in the container to form a closely spaced layer of the material on the bottom of the shallow pans that will be covered with a thin layer of the solution (next paragraph).

Open the top of the humidity control chamber and remove the two pans. Fill each pan with the saturated salt solution being sure that a small quantity of excess (undissolved) salt is also transferred to each pan. Replace the pans and seal the top of the plastic enclosure; air should enter the enclosure primarily from the front opening.

9.3.3 Priming the Scrubber

Fill the round bottom flask of the scrubber to about one third full with asbestos-free (i.e. filtered, distilled) water. Initiate the flow of ice water through the entrance and exit condensers. Adjust the variable voltage transformer on the heating mantle so that water in the round bottom flask boils and the rate at which condensate drops back into the flask from the condensers is approximately equal to one drop per second.

9.3.4 Adjusting Initial Air Flow

The air flow within the various components of the dust generator must be adjusted so that flow within the vertical elutriator will properly separate and pass only respirable particles. Based on the discussion presented in Appendix A (Section A.2.3), the proper linear flow rate in the elutriator shall be set at 0.31 cm/s, which is 5% greater than the Stokes' velocity estimated for the largest spherical, respirable particles (i.e. those with a radius of 5 μm).

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[20] Filters to be used to collect samples over the isokinetic sampling tube of the elutriator (see Section 9.4.5) must be weighed to a minimum precision of ± 0.00002 g.

[21] Note that potassium carbonate dihydrate is not the usual form of potassium carbonate sold commercially. The usual commercial forms are the anhydrous and the sesquihydrate. The dihydrate can be made by allowing either of the commercial varieties of the salt to stand in their saturated solution for some extended period of time with some temperature cycling (Berman and Kolk 1994). A week appears to be sufficient but the process can be accelerated by augmenting the temperature cycling.

Next, calculate the required volumetric air flow, V_v, within the elutriator using Equation 9-1:

$$V_v = 81.1 * V_l \quad (9\text{-}1)$$

where:

V_l is the estimated linear flow rate required to separate respirable particles (i.e. 0.31 cm/s); and

V_v is the corresponding volumetric flow rate (cm^3/s) through the elutriator.

The coefficient, 81.1, in Equation 9-1 corresponds to the cross-sectional area of the elutriator (in cm^2).

To adjust the initial flow valve settings on the pumps, first connect one of the flowmeters such that air flows directly from the exit opening at the top of the elutriator that is *not* articulated with the isokinetic sampling tube[22]. This is the exit that opens directly into the top, tapering portion of the elutriator (see Appendix A) and is labeled the "ME" opening in Figure A-1. A filter cassette containing a filter from the batch of filters to be used for the run shall be placed between the flowmeter and the pump when adjusting the flow valve on this pump.

NOTE

The easiest way to directly connect a flow line to one of the top openings of the elutriator (the ME or the IST opening) is to align the appropriate slide mechanism so that a filter mount is directly over the opening, mount a cassette *without* a filter into the mount, and connect the air flow line to the exit side of the filterless cassette (see Section A.1.6 of Appendix A). During calibration, such a line would then feed sequentially into a flowmeter, a filter-containing cassette, and a pump.

Connect a second flowmeter directly to the exit line of the scrubber (the entrance line of which should already be attached to the two side exit openings on the elutriator) and place a filter cassette containing a filter from the batch of filters to be used for the run between this flowmeter and the pump for the scrubber.

Adjust the flow control valves on both the scrubber pump and the pump to be connected to the ME opening of the elutriator so that flow in both lines are equal and that each flow is set at $0.48 * V_v$.

Connect a third flowmeter to the exit opening on the top of the elutriator that articulates with the isokinetic sampling tube. This is labeled the "IST" opening in Figure A-1.

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[22] To access this opening, it will be necessary to dismount any filter cassettes from the appropriate slide mechanism and to align one of the two openings in the appropriate slide mechanism over the desired opening in the elutriator (see Appendix A).

The flowmeter attached to the IST opening shall also be backed by a filter cassette containing a filter from the batch to be used during the run and adjust the flow in this line so that it is equal to $0.047*V_v$[23]. Due to the low flow required on this line, an auxiliary low flow valve is also attached to this pump and must be adjusted to achieve the desired flow. To optimize conditions, it may be necessary to adjust the flow control valves on the three air lines that exit the elutriator iteratively.

To prepare for a run using the dust generator, disconnect the flowmeters from the slide mechanisms over the top openings of the elutriator (i.e. the ME and IST openings) and mount filters in each of the four cassette holders on the two slide mechanisms that cover the two elutriator openings. Connect one of the air flow lines coming off of the "T" from each pump to each of the two filters on the same slide mechanism for the appropriate opening of the elutriator (i.e. the opening for which the pump had been calibrated). Then adjust each slide so that one filter cassette is aligned directly over each elutriator opening. Be sure that the valves on each "T" are configured so that flow is directed from the filter cassette that is aligned directly over the elutriator opening (see Section A.1.6 of Appendix A).

NOTE

The connections between the exit side of the filter cassettes mounted on the slide mechanisms and the flow control valves on the pumps should now be direct; there should be no second filter cassette in the line.

The flowmeter attached to the exit line of the scrubber may remain attached during a run to monitor air flow through the scrubber. If there is a desire also to monitor airflow through one or both of the filter cassettes mounted over the top openings of the elutriator during a run, flowmeters may now be attached to the downstream side of the filter cassettes (i.e. between the filter cassettes and the pumps). Due to the pressure drop across the filters, however, the readings from these flowmeters must be adjusted using the following equation to provide estimates of the true flow through each elutriator opening:

$$F_t = R_f*(P_f*T_t/P_t*T_f) \qquad (9\text{-}2)$$

where:

F_t is the true flow rate through the elutriator opening (cm/s);

R_f is the flow reading from the flowmeter (cm/s);

P_f is the pressure at the flowmeter (torr);

T_f is the *absolute* temperature at the flowmeter (^oK);

---

[23] The coefficient, 0.047, used to estimate the volumetric flow rate for air passing through the isokinetic sampler represents the fraction of the cross-sectional area of the elutriator that is subtended by the isokinetic sampling tube and therefore represents the fraction of the total flow that should pass through the tube, assuming that flow in the elutriator has been properly set.

P_t is the pressure at the elutriator opening (torr); and

T_t is the *absolute* temperature at the elutriator opening (^oK).

To use this equation, P_f and P_t will have to have been measured during flow calibration, prior to a run, using mercury manometers or other appropriate pressure measuring devices. Generally, T_t and T_f can be considered equal and will drop out of the equation. However, Equation 9-2 can also be used to adjust flow readings between calibrations and runs that are conducted on different days, such that temperatures may vary between the time during which the calibration was conducted and the time that the run is performed.[24]

9.4 DUST GENERATOR OPERATION

To prepare asbestos samples using the dust generator, load the tumbler, condition the bulk sample, begin the run, monitor the rate of dust generation, and collect appropriately loaded filters for asbestos analysis. Asbestos is also collected in the scrubber. Prior to use, be sure that the dust generator is clean (see Section 9.5).

9.4.1 Loading the Tumbler

Detach the tumbler from its drive motor and the vertical elutriator and remove it from the plastic enclosure at the bottom of the dust generator (see Appendix A). Place the tumbler on a flat surface and open the top for loading. Be sure that the tumbler is clean prior to loading.

Introduce a sample[25] by holding the sample container against the inner lip of the tumbler and tilting the container so that the sample pours smoothly into the tumbler. Move the sample container back and forth along the length of the tumbler to facilitate uniform deposition of the sample in the tumbler. When pouring is complete, tap the sample container vigorously so that the quantitative transfer is complete. The masses of samples introduced into the tumbler shall range between 50 and 80 g. Larger samples shall be homogenized and split prior to loading as described in Section 9.2.

Shake the tumbler gently to assure uniform deposition of the sample within the tumbler, which should be no more than about one third full. Be sure that the rubber gasket on the tumbler is in good repair and properly seated. Replace the gasket if it is worn. Secure the top of the tumbler with 10 screws and replace the tumbler within the plastic enclosure at the bottom of the dust generator. Reattach the elutriator entrance tube and D.C. motor to the tumbler (see Appendix A).

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[24] Because the viscosity of air is somewhat temperature dependent, when runs are to be conducted at temperatures that differ by more than a few degrees from room temperature (nominally 20° C), Equation A.10 (in Appendix A) may have to be adjusted to account for the varying viscosity (see Equation A.9) so that the correct flow regime can be established at the new temperature to assure that the elutriator of the dust generator passes only respirable particles.

[25] Samples to be introduced into the tumbler shall have been dried per Section 9.1.

9.4.2 Conditioning the Sample

Before conditioning the sample, be sure that the dust generator has been properly set up. This means, check that:

- the pans in the constant humidity chamber have been filled with saturated solution;
- filters have been mounted on each of the four cassette mounts on the slide mechanisms atop the elutriator;
- water is boiling in the scrubber;
- the air flow valves have been properly set; and
- all air lines between the dust generator, flow valves, and pumps are properly configured (see Section A.1.6 of Appendix A).

To condition the sample, turn on all pumps and begin the flow of air through the dust generator. ***DO NOT TURN ON THE TUMBLER MOTOR.*** Allow the flow of air to continue for a minimum of two hours before beginning a run. If the sample was oven dried rather than equilibrated with an appropriate salt in a desiccator (Section 9.1), the sample should be conditioned for a minimum of four hours prior to initiating a run.

9.4.3 Initiating a Run

Once the sample has been conditioned, set the tumbler drive motor to 30 rpm and turn it on. Simultaneously, move the two slide mechanisms at the top of the elutriator so that new, clean filters are now aligned over both the ME and IST openings of the elutriator. Be sure to change the valve orientations on the lines leading to the filters so that air flow is directed through the filter cassettes that are newly aligned with the elutriator openings.

Replace the filters originally aligned over the elutriator openings (but no longer aligned) with clean filters and weigh and store the old filters in labelled Petri dishes. These filters are equipment blanks. After five minutes,[26] move the sliding mechanism again to bring new, clean filters over the ME and IST openings of the elutriator. Immediately after the filters are brought out of alignment, dismount the cassette and turn the potentially dust-laden side of the filter face up before halting the flow of air through the filter (by turning the appropriate valves). Once flow from the dismounted cassettes has been halted (correspondingly, flow will have been re-directed to the cassettes that are currently aligned over the elutriator openings), replace the dismounted filters with clean filters and weigh and store the dismounted filters in labelled Petri dishes. These filters are run blanks.

As the run proceeds, record the times that air flow was started and stopped for each filter, the initial and final weights of each filter, and the identifier of each filter in a log book.

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[26] This interval is selected because, in the absence of channeling, five minutes is just less than the time over which the fastest particles are expected to reach the filter.

9.4.4 Monitoring the Rate of Respirable Dust Generation

The rate of respirable dust generation is monitored during a run by recording the weights of a set of filters that are sequentially changed out of the filter mounts over the ME opening of the elutriator at defined, regular intervals.

Initially, change the filter that is aligned over the ME opening of the elutriator at intervals of five to eight minutes. The change is accomplished by moving the slide mechanism to switch a new filter into alignment at the same time that the old filter is switched out of alignment. Immediately after the filter is brought out of alignment, dismount the cassette and turn the dust-laden side of the filter face up before halting the flow of air through this filter (i.e. by turning the appropriate valves to re-direct air flow to the filter that is newly aligned over the ME opening). Exchange the old filter for a new filter.

NOTE

Because the time during which air flow is directed through a dismounted cassette (rather than the filter that is aligned over the elutriator opening) results in a disturbance in the otherwise smooth flow of air through the elutriator, the changing of filters shall be performed expeditiously. As long as this interval is not more than a few seconds, however, studies indicate that this effect is not significant (Berman and Kolk 1994).

Along with the proper identifier, record the times during which air flow is started and halted for each filter. Weigh each filter after dismounting. Record the initial and final weights of the filter and the net weight of dust deposited on the filter (i.e. the difference between the initial and final weight).

After exchange of the first two or three filters, the interval over which dust is collected on each filter may be optimized. The ideal weight of dust to be deposited on each filter is between 0.01 and 0.03 g (Berman and Kolk 1994). Based on the rate of dust deposition on the first two or three filters, estimate the interval of time required to deposit approximately 0.02 g and exchange later filters at this rate (see Section A.2.2 of Appendix A, Equation A-8)[27]. However, it is important that time be adjusted so that no more than 0.03 g be deposited on each filter because the possibility that a portion of the deposit accidently drops from the filter increases as the weight of the deposit on the filter increases.

NOTE

Until there is a need for generating filters for asbestos analysis (see Section 9.4.5), the filters that get aligned over the IST opening of the elutriator need to be changed only one fifth to one tenth as often as the filters over the ME opening of the elutriator. These filters shall be changed at this lower rate, however, to prevent the potential for a heavy deposit to drop off of the filter and fall back into the elutriator.

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[27] At the beginning of a run the rate of dust deposition on the filters has been observed to be nearly constant with time.

Continue the run at 30 rpm (with continuing exchange of filters) for approximately two hours; this time interval has generally been observed as sufficient to define the rate of respirable particle release at this rotation rate (Berman et al 1994a). Generally, the plot of the release of respirable dust versus time at 30 rpm shows almost no curvature (see Section 11.2).

After completing the run at 30 rpm, select a new, higher rotation rate to continue the run. Generally, the new rotation rate selected shall be 60 rpm, unless the rate of release at 30 rpm was noticeably low in comparison with prior runs on other samples, in which case 90 or 120 rpm shall be used.

NOTE

Use of the highest rotation rates should generally be avoided, unless there is compelling evidence for their efficacy, because they tend to facilitate the transport of non-respirable particles from the tumbler into the bottom of the elutriator and, if such transport is heavy, this may affect results (see Section A.1.3 of Appendix A).

Continue the run at the higher rate of rotation by collecting a minimum of eight additional dust-laden filters. The same procedures outlined above should be continued for collecting data during the run at this higher rotation rate except that the interval between the exchange of filters must be adjusted downward to assure that deposits on these latter filters do not exceed 0.03 g (see Section A.2.2 of Appendix A, Equation A-8, but note that the rate constant, k, is dependent on the rotation rate for the tumbler so that Equation A-8 cannot be extrapolated across runs).

When the run is complete, turn off the tumbler motor but allow the air flow to continue for ten or fifteen additional minutes to empty the elutriator. Be sure to continue the exchange of filters, if necessary to prevent overloading. The air flow pumps may now be shut off.

9.4.5 Generating Appropriately Loaded Filters for Asbestos Analysis

The primary purpose for collecting dust on filters mounted over the exit of the isokinetic sampling tube of the elutriator (the opening labeled "IST" in Figure A-1) is to obtain samples suitable for asbestos analysis using a direct transfer technique, although use of an indirect transfer technique is not precluded. This is an option built into the design of the dust generator as an alternative to preparation of a specimen for asbestos analysis using the liquid from the scrubber, which necessarily mimics an indirect transfer technique because (in the scrubber) the asbestos is captured and suspended in water.

Collect filters for asbestos analysis near the end of each of the two runs (i.e. one run at each of two rotational speeds for the tumbler) that is described in the previous section of this chapter. Filters to be used for asbestos analysis shall be collected at the end of the runs both because this will be the period when the rate of asbestos emission is the lowest and because sufficient time will have elapsed over each run to allow a steady-state distribution of particle sizes to have developed in the dust traversing the elutriator. At the beginning of a run, only the smallest (fastest) respirable particles reach the filters and it takes time for the larger (slower) respirable particles in the air stream to begin reaching the filters in numbers that are proportional to their rate of emission from the tumbler. It takes several tens of minutes for transport of a steady state distribution to develop.

Collect multiple filters during each run that bracket the estimated time during which an optimal loading for analysis of a directly prepared specimen is expected to be achieved. Mount, exchange, dismount, weigh, record, and store filters precisely in the manner described in Section 9.4.4.

Estimate the time required for achieving an optimum loading as follows. The optimal mass loading on a filter to be prepared by a direct transfer technique lies between 1 and 10 μg (see, for example, Berman and Chatfield 1990). Assume a target of 5 μg. However, this may have to be adjusted based on experience with the dust generator. Equation A-7 (Appendix A) can then be re-arranged to estimate the time required to collect 5 μg (or some other defined mass) of dust:

$$\Delta t = \Delta M_f / 0.047 * k * M_s \qquad (9\text{-}3)$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" after the beginning of the run, but it is assumed constant over the short interval of time "Δt" (g);

ΔM_f is the mass of respirable dust collected on a filter over the IST opening during the short time interval "Δt" (as indicated above, assume a target of 5 μg or 5×10^{-6} g);

Δt is a short time interval (no more than several minutes) during which the release of dust is being estimated (s); and

k is the first-order rate constant for the release that is derived from the dust measurements collected during the run (s^{-1}).

The mass of respirable dust remaining in the sample during the time interval of interest (K_s) is estimated using a rearrangement to Equation A-2 (Appendix A):

$$M_s = M_o * \exp(-kt) \qquad (9\text{-}4)$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" (g);

M_o is the mass of respirable dust in the sample at the start of the run (i.e. at time t = 0) (g).

t is the time from the start of the run to the beginning of the time interval "Δt" (s); and

k is the first-order rate constant for the release (s^{-1}).

Based on the recently completed pilot study for this method (Berman et al. 1994a), a typical rate constant for dust emission from the tumbler is 0.004 min^{-1} (6.7×10^{-5} s^{-1}). Results from this study also suggest that the range in respirable dust content likely to be encountered for samples typically run using the dust generator may vary between 0.5% and 2%. Therefore, given a typical sample mass of 70 g, M_o for the 30 rpm run likely ranges between approximately 0.17 and 0.68 g. Given that a typical 30 rpm run lasts for approximately 3 hours (1.1×10^4 s) and substituting these values for M_o, k, and t into Equation 9-4, and then substituting the subsequent estimate of M_s into Equation 9-3, it appears that between 2.3 and 9 seconds would be required at the end of the 30 rpm run to collect 5 µg of material on a filter loaded over the isokinetic sampler.

Similarly, assuming that the initial mass of respirable dust in a sample at the beginning of a 60 rpm run, $M_{o\text{-}60}$, is equal to the remaining mass at the end of the 30 rpm run (i.e. the "M_s" calculated above), remembering that a 60 rpm run typically lasts 2 hours (7.2×10^3 s), and once again noting the typical value for k indicated above, Equation 9-4 is used to estimate an appropriate M_s for the end of a 60 rpm run. Substituting this new value into Equation 9-3, it appears that between 4 and 15 seconds may be required to collect 5 µg of material on a filter that is loaded over the isokinetic sampler at the end of a 60 rpm run.

Given the above, to properly bracket the optimal loading for a filter to be employed for asbestos analysis using a (direct transfer technique), collect filters over the IST opening of the elutriator that are exposed for periods of 3, 10, and 20 seconds (at both the end of the 30 rpm run and the end of the 60 rpm run).

9.4.6 Obtaining Asbestos Samples from the Scrubber

At the end of all runs for a particular sample (after all pumps have been shut off), turn off the heating mantle to the scrubber and let it cool for 10 to 15 minutes before discontinuing the flow of ice water to the condensers. Disconnect the outlet lines from the elutriator to the scrubber at the elutriator. Samples shall be extracted from the scrubber *expeditiously* to minimize losses to the walls of the glassware and to facilitate cleaning.

To minimize loss, before disconnecting the transfer lines and condensers from the round-bottom flask of the scrubber, pour approximately 100 ml of asbestos-free (filtered, distilled) water down the exit condenser and another 100 ml down the transfer lines and entrance condenser. Such rinsing should be performed in multiple stages, approximately 20 ml at a time. Swirl each condenser (and the transfer lines) as the water drains into the flask. Tap each condenser (and the transfer lines) several times after rinsing to assure a reasonably quantitative transfer.

Detach and remove the round-bottom flask from the condensers and its stand and pour the contents of the flask into a clean, pre-weighed, wide-mouthed 1 L plastic container. Rinse the round bottom flask several times with additional asbestos-free water to assure a quantitative transfer of any residual solids. Reweigh the container and record the net weight as the total weight of suspension. If necessary, the sample may then be stored at ice temperature until it can be prepared. However, preparation shall not be delayed for more than 48 hours.

Immediately prior to filter preparation, add 1.5 g/L of sodium hexametaphosphate to the suspension in the plastic container. Shake the suspension vigorously and divide it

approximately evenly into two (or, if necessary, three) 500 ml Erlenmeyer flasks. Place the flasks on a laboratory shake table for approximately three hours. Quickly re-combine the contents of the two (or three) flasks into a clean, plastic container (with minimal flushing) and place the container in a sonicator. Sonicate the suspension for approximately 1 minute (with the power of the sonicator set at no more than 0.1 W/ml). Withdraw one ml with a disposable pipette from the center of the volume of the suspension in the plastic container and dilute this with asbestos-free water to 100 ml in a clean, volumetric flask.

The mass of respirable dust collected in the scrubber should be equal to the sum of the cumulative mass of dust measured on the filters collected above the ME opening of the elutriator over the entire run(s) during which the scrubber suspension was collected. Use this estimated mass (and account for the 100-fold dilution performed as described in the last paragraph) to estimate the size of aliquots required to produce filterable suspensions containing: 0.5, 2, and 5 µg of respirable material. Dilute each aliquot to a minimum total volume of 20 ml and filter each aliquot in the manner described in Sections 10.34 and 10.35 of the ISO Method for the determination of asbestos in air using an indirect transfer technique (Chatfield 1993)[28].

The filtered aliquots shall all be prepared as described in Section 10.1 and scanned briefly at low magnification in the TEM to select the optimally loaded specimen for detailed analysis (see Section 11.1.2).

9.5 CLEANING THE DUST GENERATOR

The dust generator is designed for quick and easy assembly and disassembly to facilitate cleaning. Most of the joints are simple friction couplings or ring clamp couplings. To clean the dust generator, disconnect and disassemble the tumbler, remove the bottom cup and dust collector system from the elutriator, decouple the two halves of the elutriator tube, disassemble the slide mechanisms of the dust collector and disconnect the transfer lines to the scrubber. The metal pieces of the dust generator may then be washed with biodegradable detergent, rinsed with asbestos-free water, sonicated briefly, and rinsed again. The pieces may then be left to dry in room air or may be dried with a forced, HEPA-filtered air stream.

The glassware of the scrubber shall also be washed with biodegradable detergent and asbestos-free water, rinsed liberally, and dried in room air or dried with a forced, HEPA-filtered air stream. It is recommend that new transfer lines between the elutriator and the scrubber (constructed of 1.00 in. i.d. Tygon tubing) be cut and installed after each cleaning.

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[28] As indicated in Chatfield (1993), filters to be employed for filtering scrubber suspensions are of a different type than those employed in dust generator mounts. Filters used to filter scrubber suspension are to be the 0.22 µm pore size variety

10.0 PREPARATION OF SPECIMEN GRIDS FOR TEM ANALYSIS

For Superfund applications of this method, asbestos analysis of all samples prepared using the dust generator are to be performed on specimen grids prepared from aliquots of the scrubber suspension. In addition, for a minimum of a subset of 5% (preferably 10%) or 10 samples (whichever is greater), asbestos analysis is also to be performed on specimen grids prepared by a direct transfer technique from filters collected over the IST opening of the elutriator (i.e. the opening over the isokinetic sampling tube). These analyses are then paired with the analyses of asbestos from scrubber suspension collected during the same runs to provide a link between samples prepared by each technique.

The primary reason for preparing 100% of samples from the scrubber suspension is to facilitate identification of distinctions in sample characteristics; samples prepared in this manner are expected to exhibit the best precision among the options for this method. At the same time, when comparing results to published slope factors, the apparent need for normalizing asbestos analyses to counts derived from directly prepared specimens (see, for example, Berman and Crump 1989) is satisfied by providing a subset of samples prepared both ways to allow a regression to be performed linking results from the scrubber suspension samples to specimens prepared by a direct technique. As indicated previously (Section 2.1.3), the recommended procedure is based on a compromise allowing optimum precision for distinguishing among relative measurements (and relative risks) while excepting a small reduction in the precision of estimates of absolute risk.[29]

10.1 PREPARATION OF SPECIMEN GRIDS FROM FILTERED ALIQUOTS OF THE SCRUBBER SUSPENSION

Filters generated from aliquots of the scrubber suspension (as described in Section 9.4.6) shall be prepared using the direct transfer technique that is described in Sections 10.5 of the ISO Method (Chatfield 1993). As indicated previously, multiple aliquots representing a sequence of dilutions are to be prepared to allow selection of the optimally loaded filters (and corresponding set of specimen grids) for final, detailed analysis.

From each filter, prepare a minimum of three specimen grids: one from near the center of the filter, one from a location that is half the distance between the center and the outer edge, and one from near the outer edge of the filter.

10.2 SPECIMEN GRID PREPARATION FROM FILTERS COLLECTED OVER THE IST OPENING OF THE ELUTRIATOR

Although this method specifies that filters collected over the IST opening of the elutriator shall be prepared using a direct preparation technique, an indirect preparation technique is also described, as an option for non-Superfund applications.

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[29] For other applications of this method, options might include preparation of 100% of samples from filters collected over the IST opening of the elutriator and/or preparation of filters collected over this opening using an indirect transfer technique. The method is designed to be flexible.

10.2.1 Specimen Grid Preparation Using a Direct Transfer Technique

Filters collected over the IST opening of the elutriator (as described in Section 9.4.5) shall be prepared using the direct transfer technique that is described in Section 10.5 of the ISO Method (Chatfield 1993). As indicated previously, sections of multiple filters representing a range of loadings are to be prepared to allow selection of the optimally loaded specimen grids for final, detailed analysis.

From each filter that has been collected over the IST opening, prepare four specimen grids from locations on the filter that are each separated by 90° radially. Select two of the locations (from opposing sides of the filter) at points that are about two thirds of the distance from the center to the edge of the filter. The remaining two locations shall be selected at points that are about one third of the distance from the center to the edge of the filter. Such an arrangement will eliminate any effects potentially associated with a linear gradient across the filter that may develop due to the brief time over which the filters are exposed to air flow from the elutriator and, consequently, the potentially significant time during which the filter is being slid in and out of alignment.

10.3 Specimen Grid Preparation Using an Indirect Transfer Technique

As an option to the procedure described in Section 10.2.1 above (for non-Superfund applications *only*), filters collected over the IST opening of the elutriator (as described in Section 9.4.5) may also be prepared using the indirect transfer technique that is described in Sections 10.3 to 10.5 of the ISO Method (Chatfield 1993). For this option, multiple sections of the most highly loaded filter obtained from the dust generator shall be prepared using a range of dilutions to allow selection of the optimally loaded specimen grids for final, detailed analysis.

11.0 PROCEDURES FOR ASBESTOS AND DUST ANALYSIS

11.1 PROCEDURES FOR ASBESTOS ANALYSIS

Specimen grids prepared as described in Chapter 10 are to be analyzed using transmission electron microscopy (TEM). Follow the procedures for analysis described in the ISO Method (Chatfield 1993) including procedures for:

- examining specimen grids to determine acceptability for analysis;
- structure counting by TEM (except determination of the stopping point);
- structure morphological classification;
- structure mineralogical identification; and
- blank and quality control determinations.

The stopping points for the analyses conducted in support of this method are a function of the required sensitivity for the method and are defined in Sections 11.1.1 and 11.1.2 below.

Begin by examining one of each set of specimen grids derived for a defined loading from a particular run of the dust generator and select the optimally loaded set for analysis. Use the criteria for determining the acceptability of specimen grids (from the ISO Method) to define optimal loading.

When performing detailed analysis, be sure to distribute asbestos counts evenly over the entire set of specimen grids prepared from a particular filter at a defined (optimal) loading. Record the morphology and mineral type of asbestos structures as described in the ISO Method (Chatfield 1993). Also as described in the ISO Method, complete separate scans for counts of total structures and, at lower magnification, for counts of structures longer than 5 μm.

11.1.1 Analysis of Specimen Grids Prepared from Filters Collected Over the IST Opening of the Elutriator

Prior to initiating a detailed analysis of specimen grids, the stopping rules for the analysis must be defined. Assuming these specimen grids have been prepared using a direct transfer technique (as discussed in Section 10.2), define the stopping rules for the detailed analysis as follows.

First, calculate the maximum number of grid openings that will have to be scanned during the analysis from the relationship:

$$N_{go} = S_d * A_f * \%RD / (S_{smpl} * A_{go} * 100 * \Delta M_f) \qquad (11\text{-}1)$$

where:

N_{go} is the maximum number of grid openings to be scanned;

S_d is the number of structures required to define detection using the analysis (defined here as 1);

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9;

S_{smpl} is the required analytical sensitivity for the method (s/g);

A_{go} is the area of a single grid opening (mm^2); and

ΔM_f is the mass of respirable dust collected on the filter from which the specimen grids were prepared. It is defined using Equation A-7 of Section A.2.2 of Appendix A (g).

The following are typical values for the above parameters:

S_d = 1;
S_{smpl} = 5×10^7 (for total structures) or
= 3×10^6 (for structures longer than 5 µm)[30];
A_f = 382 mm^2;
A_{go} = 8.1×10^{-3} mm^2;
%RD = between 0.5% and 2%[31]; and
ΔM_f = 5 µg [32];

Given the above values, it is estimated using Equation 11-1 that between 1 and 4 grid openings will typically need to be scanned to derive a count of total structures. However, a minimum of 4 grid openings shall be scanned during any analysis. Based on the above, similarly, between 15 and 62 grid openings will typically need to be scanned to derive a count of structures longer than 5 µm.

The number of grid openings to be scanned for specific analyses shall be determined by substituting case-specific values for the above listed parameters into Equation 11-1.

Stop the counting, characterization, identification, and recording of asbestos structures on a particular analysis when one of the following obtains:

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[30] See Section 2.1.1.

[31] This is the range of values observed for a diverse variety of samples tested during the pilot study for this method (Berman et al. 1994a).

[32] See Sections 9.4.5 and A.2.2 of Appendix A.

- the scan is completed for the grid opening on which the 50th asbestos structure is counted; or

- either 4 grid openings or the maximum number of grid openings (estimated as defined above), whichever is greater, are scanned completely.

These rules are to be applied separately to the scan for total structures and the scan for long structures (i.e. longer than 5 μm) that are described in the ISO Method.

11.1.2 Analysis of Specimen Grids Prepared from Filtered Scrubber Suspension

Prior to initiating a detailed analysis of specimen grids, the stopping rules for the analysis must also be defined in this case. Given that these specimen grids have been prepared as described in Section 10.1, define the stopping rules for the detailed analysis as follows.

First, calculate the maximum number of grid openings that will have to be scanned during the analysis from the relationship:

$$N_{go} = S_d * A_f * \%RD * DF / (S_{smpl} * A_{go} * 100 * M_{scrbr}) \quad (11\text{-}2)$$

where:

N_{go} is the maximum number of grid openings to be scanned;

S_d is the number of structures required to define detection using the analysis (defined here as 1);

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9;

S_{smpl} is the required analytical sensitivity for the method (s/g);

A_{go} is the area of a single grid opening (mm^2);

DF is the dilution factor by which the scrubber suspension had to be diluted to prepare specimen grids that are suitably loaded for analysis; and

M_{scrbr} is the mass of respirable dust collected in the scrubber suspension during the run.

The dilution factor, DF, is simply the product of the individual dilution factors for the two sequential dilutions performed to derive the final volume that is ultimately filtered (per the procedure defined in Section 9.4.6):

$$DF = (V_s/V_{a1})*(V_d/V_{a2}) \quad (11\text{-}3)$$

where:

V_s is the volume of the initial scrubber suspension (which is estimated from the recorded weight of the supernatant assuming a density of 1 g/cm^3) (ml);

V_{a1} is the volume of the first aliquot collected from the scrubber suspension for further preparation (the default defined in Section 9.4.6 is 1 ml);

V_d is the volume into which the first aliquot from the suspension is diluted (the default defined in Section 9.4.6 is 100 ml); and

V_{a2} is the volume of the final aliquot collected from V_d that is ultimately filtered for preparation of the optimally loaded specimen grids (ml).

The mass of respirable dust collected in the scrubber suspension, M_{scrbr}, is derived from the cumulative mass of dust collected during the run on filters that are mounted over the ME opening of the elutriator (defined below as M_f). These differ by the ratio of the air flow into the filters and the air flow into the scrubber:

$$M_{scrbr} = M_f*(F_s/F_c) \quad (11\text{-}4)$$

where:

M_{scrbr} is the mass of respirable dust collected in the scrubber during the run (g);

M_f is the cumulative mass of dust collected on the filters (mounted over the ME opening of the elutriator) during the same run (calculated from Equation A-4 of Section A.2.1 of Appendix A);

F_s is the volumetric air flow rate into the scrubber (cm^3/s); and

F_c is the volumetric air flow rate into the filters over the ME opening of the elutriator (cm^3/s).

Assuming air flow in the elutriator is setup as described in Section 9.3.4, F_s and F_f are equal so that M_{scrbr} simply equals M_f.

Given the typical values for the corresponding parameters provided in Section 11.1.1, assuming a typical value for DF [33] of between 2 x 10^4 and 4 x 10^4, and selecting a range of typical values for M_f (i.e. between 0.1 and 0.2 g) from among the range observed during the pilot study (Berman et al. 1994a), it is estimated using Equation 11-2 that between 1 and 8 grid openings will need to be scanned to derive counts of total structures. Similarly, it is estimated that between 8 and 133 grid openings will need to be scanned to derive counts of long structures (i.e. longer than 5 μm).

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[33] This assumes typical values for M_f (between 0.1 and 0.2 g) from among the range of values observed among diverse samples during the pilot study. (Berman and Kolk 1994) and further assumes that the dilution factor is selected so as to produce a loading of 5 μg on the filter.

Determine the actual number of grid openings required for a specific analysis by substituting case-specific values for the above parameters into Equations 11-2, 11-3, and 11-4.

Stop the counting, characterization, identification, and recording of asbestos structures on a particular analysis when one of the following obtains:

- the scan is completed for the grid opening on which the 50th asbestos structure is counted; or
- either 4 grid openings or the maximum number of grid openings (estimated as defined above), whichever is greater, are scanned completely.

These rules are to be applied separately to the scan for total structures and the scan for long structures (i.e. longer than 5 μm) that are described in the ISO Method (Chatfield 1993).

11.2 EVALUATING THE RATE OF RELEASE OF RESPIRABLE DUST

The rate of release of respirable dust from a sample prepared using the dust generator is estimated from measurements of the mass of dust collected over time on the set of filters mounted over the ME opening of the elutriator. The measurements used specifically are from those filters that are collected while the tumbler is operating at the *highest* rotation rate employed for the sample (see Section A.2.1 of Appendix A).

Begin by plotting the cumulative mass collected on the filters as a function of time. To derive the cumulative mass for a particular time interval, add the mass of dust measured on the filter collected from that time interval to the sum of the masses measured on the set of filters collected earlier in the run. Typical curves are depicted in Figures 11-1 and 11-2. Next, calculate the cumulative mass released from the sample over time from the cumulative mass collected on filters over time using the relationship developed in Section A.2.1 of Appendix A:

$$M_r = 2.1 * M_f \qquad (11\text{-}5)$$

where:

M_r is the cumulative mass of dust released from a sample between the start of a run and time "t" (g); and

M_f is the cumulative mass collected on filters[34] between the start of a run and time "t" (g).

Equation 11-5 is appropriate to use to relate the mass of dust collected on filters to the mass released from the sample when air flow in the dust generator is setup as indicated in Section 9.3.4. If different air flow conditions are established for a particular experiment, the relationship between M_r and M_f will have to be derived using Equation A-4 from Appendix A.

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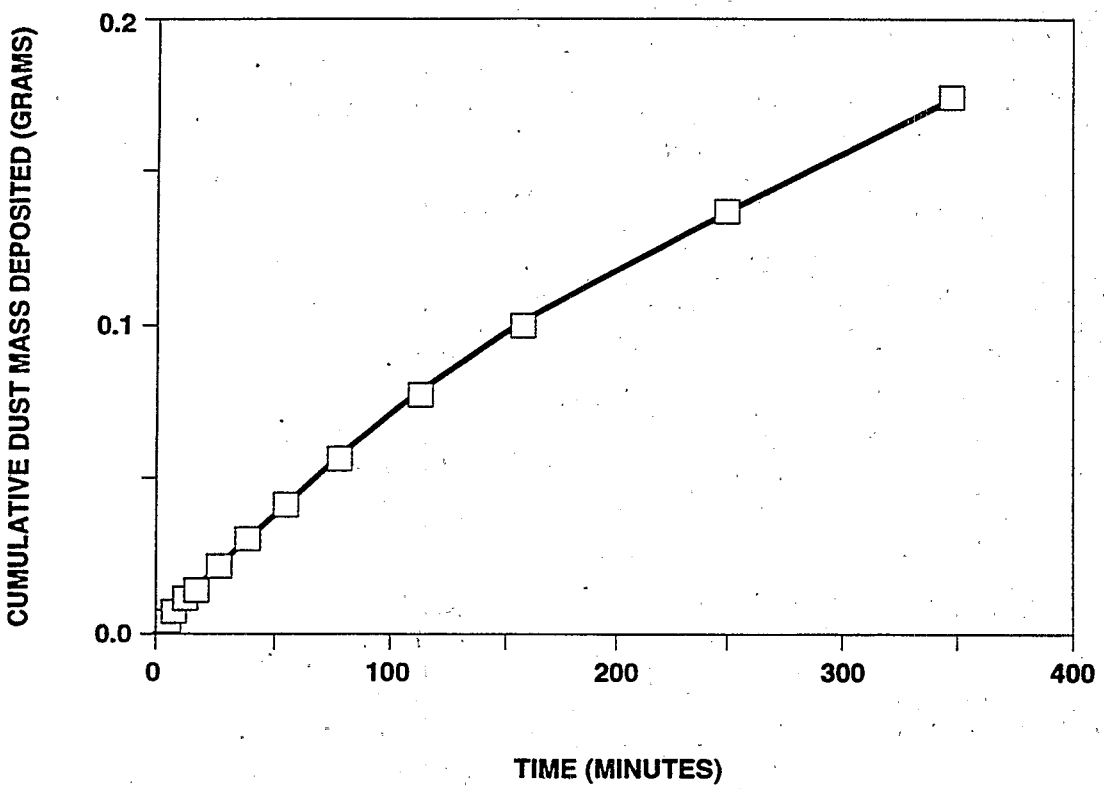
[34] These are the filters that are mounted over the ME opening of the elutriator.

FIGURE 11-1

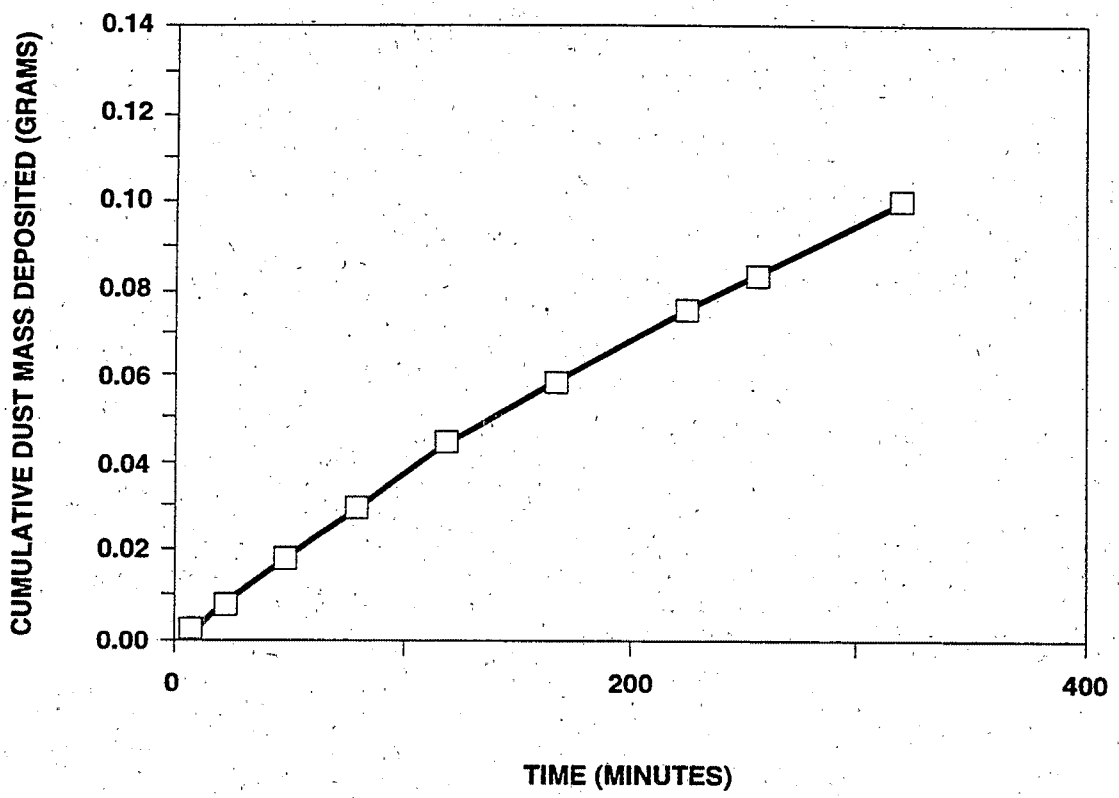
TYPICAL CUMULATIVE MASS RELEASE VERSUS TIME CURVE FOR 30 RPM RUN[a]



[a]FROM THE 30-RPM RUN ON SAMPLE 1-100, WHICH WAS COMPLETED ON 8/27/93 DURING THE PILOT STUDY (BERMAN ET. AL. 1994a).

FIGURE 11-2

TYPICAL CUMULATIVE MASS RELEASE VERSUS TIME CURVE FOR 60 RPM RUN[a]



[a]FROM THE 60-RPM RUN ON SAMPLE 1-100, WHICH WAS COMPLETED ON 8/27/93 DURING THE PILOT STUDY (BERMAN ET. AL. 1994a).

The total mass of dust in the sample at the beginning of the run must next be estimated using the relationship developed in Appendix A. Based on the relationship (see Section A.2.1):

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad (11\text{-}6)$$

where:

M_o is the mass of dust in the sample at the start of the run (g);

k is the first-order rate constant for the release of dust from the sample (s^{-1}); and

t is the time since the start of the run (s);

a plot of $\ln(M_o - M_r)$ versus t should be a straight line with a slope equal to the rate constant for the release of dust from the sample and an intercept equal to the natural logarithm of the mass of dust in the sample at the start of the run. Derive estimates of "M_o" and "k" by programming Equation 11-6 into a spreadsheet and running a regression[35].

Input a range of guesses for the value of M_o into the spreadsheet and run a regression to fit a value for k and to calculate a value for the regression coefficient, "r^2" for each value of M_o. Plot the regression coefficient, "r^2" as a function of M_o. An example of such a plot is presented in Figure 11-3. The value of M_o that provides the fit with the largest regression coefficient (i.e. with r^2 closest to 1) shall be reported as the correct value for the mass of dust in the sample at the start of the run and shall be reported with the corresponding k value as the estimated rate constant for dust release from the sample during the run.

11.3 DETERMINING THE CONTENT OF RESPIRABLE DUST

To determine the mass percent of respirable dust in the original sample, first determine the total mass of respirable dust in the sample *at the start of a run* for the last run completed on the sample, which is derived as described in the last section.

Because the dust generator run analyzed as described in Section 11.2 will generally have been preceded by a run with the tumbler speed set at 30 rpm (see Section 9.4.4), to estimate the total mass of dust present in the sample, it is necessary to include the mass released during this first run.

Sum the masses of dust measured on each of the filters collected during the 30 rpm run and designate this sum, "M_{f30}", which is the cumulative mass of dust collected during the 30 rpm run. Using the following equation, estimate the total mass of dust *released* from the sample during the 30 rpm run, "M_{r30}," based on the mass of dust *collected* during that run (see Section A.2.1 of Appendix A):

$$M_{r30} = 2.08 * M_{f30} \qquad (11\text{-}7)$$

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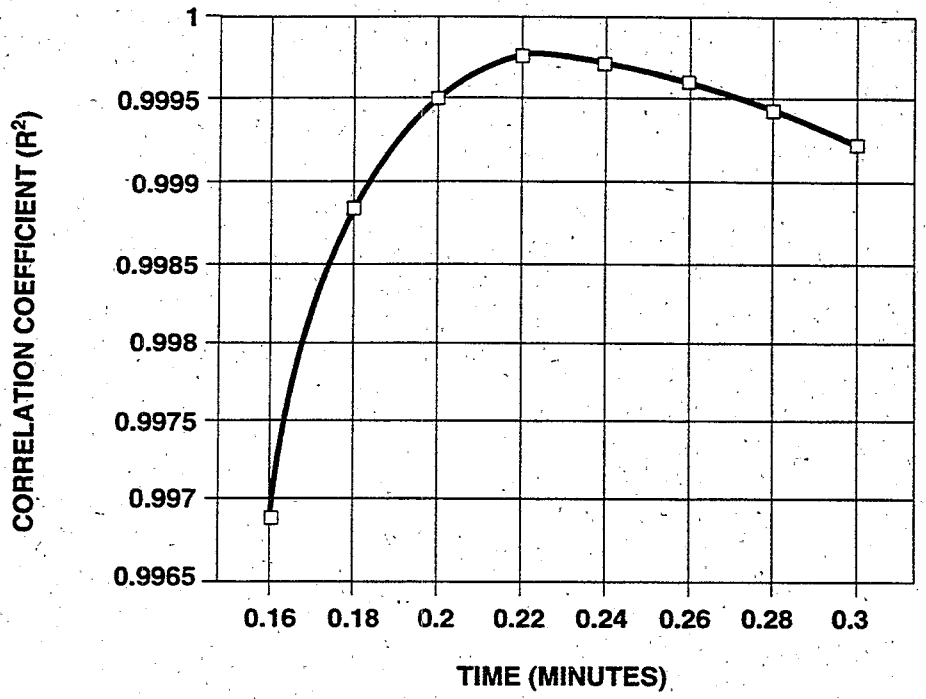
[35] Any of several commercial spreadsheet programs (including, for example, LOTUS™) contain the necessary capabilities and may be employed to derive optimum values for "M_o" and "k."

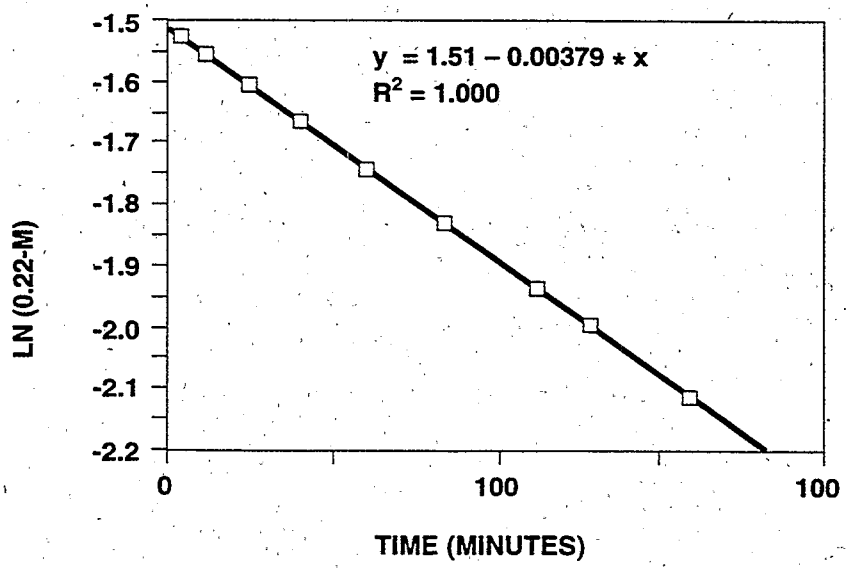
FIGURE 11-3

ILLUSTRATION OF THE OPTIMIZATION OF THE ESTIMATE OF INITIAL MASS "M_0"

A. TYPICAL PLOT OF CORRELATION COEFFICIENT VERSUS INTITIAL MASS FOR A DUST GENERATOR RUN[a]



B. TYPICAL PLOT OF THE OPTIMIZED FIRST ORDER RATE EQUATION FOR A DUST GENERATOR RUN[a]



[a]FROM THE 60-RPM RUN ON SAMPLE 1-100, WHICH WAS COMPLETED ON 8/27/93 DURING THE PILOT STUDY (BERMAN ET. AL. 1994a).

Calculate the total mass of dust originally present in the sample, M_{tot}, by summing the mass released during the 30 rpm run with the mass of dust estimated to have resided in the sample at the beginning of the higher rpm run, M_o (this is equal to the mass of dust remaining in the sample at the end of the 30 rpm run). M_o will have been derived as described in Section 11.2:

$$M_{tot} = M_{r30} + M_o \quad (11\text{-}8).$$

Estimate the mass percent of respirable dust in a sample as follows:

$$\%RD = 100*M_{tot}/M_{sample} \quad (11\text{-}9)$$

where:

%RD is the mass percent of respirable dust in the sample (%); and

M_{sample} is the mass of the original sample placed in the tumbler (g).

11.4 DETERMINING THE CONTENT OF ASBESTOS

The concentration of asbestos in a sample is determined differently depending on whether asbestos is determined from sampling grids prepared from filters collected over the IST opening of the elutriator or from scrubber suspension.

NOTE

For samples originally containing a significant fraction of coarse material (see Section 8.4), the concentration of asbestos reported as a function of the mass of the sample (specified in this section) must be adjusted for the quantity of coarse material originally measured in the matrix sampled in the field before it can be considered representative of that matrix. A procedure for adjusting asbestos concentrations to account for the coarse fraction of an environmental matrix is presented in Section 11.4.3.

11.4.1 Based on Directly Prepared Filters Collected Over the IST Opening of the Elutriator

Procedures for determining the concentrations of asbestos structures in a sample differ slightly depending on whether the structures of interest are longer or shorter than 5 μm.

Structures that are shorter than 5 μm in length are derived *only* from the high magnification scan of an analysis. Calculate and report the concentration of short asbestos in the original sample based on the counts of asbestos structures that are derived as defined in Section 11.1.1 using the following relationship:

$$C_{smpl} = S_{ch} * A_f * \%RD / (N_{goh} * A_{go} * 100 * \Delta M_f) \quad (11\text{-}10)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{ch} is the number of structures (of the defined size range or type of interest) counted during the high magnification scan of the analysis;

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9 (%);

N_{goh} is the number of grid openings scanned during the high magnification scan of the analysis;

A_{go} is the area of a single grid opening (mm^2); and

ΔM_f is the mass of respirable dust collected on the filter from which the specimen grids were prepared. It is defined using Equation A-7 (see Section A.2.2 of Appendix A) (g).

Structures that are longer than 5 µm in length are derived from combined counts collected during both the high and low magnification scans of an analysis. Calculate and report the concentration of long asbestos in the original sample based on the counts of asbestos structures that are derived as defined in Section 11.1.1 using the following relationship:

$$C_{smpl} = (S_{ch} + S_{cl}) * A_f * \%RD / [(N_{goh} + N_{gol}) * A_{go} * 100 * \Delta M_f] \quad (11\text{-}11)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{cl} is the number of structures (of the defined size range or type of interest) counted during the low magnification scan of the analysis; and

N_{gol} is the number of grid openings scanned during the low magnification scan of the analysis.

Thus, for long structures, it is the total counts of structures observed over both the high and low magnification scans and the total area scanned (over both the low and high magnification scans) that are used to determine concentration.

As an option, asbestos concentrations may also be reported as a function of the mass of respirable dust in a sample using the following relationships. For short structures, use:

$$C_{dust} = S_{ch} * A_f / (N_{goh} * A_{go} * \Delta M_f) \quad (11\text{-}12)$$

where:

C_{dust} is the concentration of asbestos structures (of any defined size range or type) in the respirable dust of the sample (s/g_{dust}); and all other parameters are defined as described above.

Similarly, for long structures, use:

$$C_{dust} = (S_{ch} + S_{cl}) * A_f / [(N_{goh} + N_{gol}) * A_{go} * \Delta M_f] \quad (11\text{-}13)$$

11.4.2 Based on Specimens Prepared from Scrubber Water

As described in Section 11.4.1, procedures for determining the concentrations of asbestos structures in a sample differ slightly depending on whether the structures of interest are longer or shorter than 5 μm.

Structures that are shorter than 5 μm in length are derived only from the high magnification scan of an analysis. Calculate and report the concentration of short asbestos in the original sample based on the counts of short asbestos structures that are derived from the scrubber suspension as defined in Section 11.1.2 using the following relationship:

$$C_{smpl} = S_{ch} * A_f * \%RD * DF / (N_{goh} * A_{go} * 100 * M_{scrbr}) \quad (11\text{-}14)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{ch} is the number of structures (of the defined size range or type of interest) counted during the high magnification scan of the analysis;

A_f is the total area of the filter from which the specimen grids were prepared (mm^2);

%RD is the mass percent of respirable dust in the sample and is defined using Equation 11-9 (%);

N_{goh} is the number of grid openings scanned during the high magnification scan of the analysis;

A_{go} is the area of a single grid opening (mm^2);

DF is the dilution factor by which the scrubber suspension had to be diluted to prepare specimen grids for analysis (derived as defined in Equation 11-3); and

M_{scrbr} is the mass of respirable dust collected in the scrubber suspension during the run (derived as defined in Equation 11-4).

Structures that are longer than 5 μm in length are derived from combined counts collected during both the high and low magnification scans of an analysis. Calculate and report the concentration of long asbestos in the original sample based on the counts of long asbestos structures that are derived from scrubber suspension as defined in Section 11.1.2 using the following relationship:

$$C_{smpl} = (S_{ch} + S_{cl})*A_f*\%RD*DF/[(N_{goh} + N_{gol})*A_{go}*100*M_{scrbr}] \quad (11\text{-}15)$$

where:

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the original sample (s/g);

S_{cl} is the number of structures (of the defined size range or type of interest) counted during the low magnification scan of the analysis; and

N_{gol} is the number of grid openings scanned during the low magnification scan of the analysis.

As an option for analysis of the scrubber suspension, asbestos concentrations may also be reported as a function of the mass of respirable dust in a sample using the following relationships. For short structures, use:

$$C_{dust} = S_{ch}*A_f*DF/(N_{goh}*A_{go}*M_{scrbr}) \quad (11\text{-}16)$$

where:

C_{dust} is the concentration of asbestos structures (of any defined size range or type) in the respirable dust of the sample (s/g_{dust}); and all other parameters are defined as described above.

Similarly, for long structures, use:

$$C_{dust} = (S_{ch} + S_{cl})*A_f*DF/[(N_{goh} + N_{gol})*A_{go}*M_{scrbr}] \quad (11\text{-}17)$$

11.4.3 Procedure for Adjusting Asbestos Concentrations to Account for the Presence of Coarse Material in the Sampled Matrix

As indicated in Section 8.4, due to the need to incorporate field data into this calculation, a formal protocol designating who is to perform this calculation and how that individual is to obtain the needed field information must be defined at the start of a study using this method.

To provide a representative measure of the concentration of asbestos in an environmental matrix in which significant coarse material is found (i.e. more than 10% by weight), first derive an appropriate coarseness adjustment factor:

$$CF = M_{fine}/(M_{fine} + M_{coarse}) \quad (11\text{-}18)$$

where:

CF is the coarseness adjustment factor for a particular sample (dimensionless);

M_{fine} is the mass of fine material measured immediately after sieving the sample in the field (g); and

M_{coarse} is the mass of coarse material measured immediately after sieving the sample in the field (g).

Then, to determine the concentration of asbestos in the environmental matrix that was sampled, perform the following adjustment:

$$C_{mtrx} = CF^{*}C_{smpl} \quad (11\text{-}19)$$

where:

CF is the coarseness adjustment factor for a particular sample (derived as defined above);

C_{mtrx} is the concentration of asbestos structures (of a defined size range or type) in the field matrix sampled (s/g); and

C_{smpl} is the concentration of asbestos structures (of a defined size range or type) in the sample sent to the laboratory (s/g). This is determined as described in Sections 11.4.1 and 11.4.2 above.

12.0 PERFORMANCE CHARACTERISTICS AND QUALITY CONTROL/QUALITY ASSURANCE REQUIREMENTS

12.1 METHOD PERFORMANCE

The method defined in this document achieves the target performance requirements defined in Section 2.1 at a cost that should be competitive with other procedures that might be designed to produce comparable information (Section 2.4).

12.1.1 Analytical Sensitivity

As indicated in Section 11.1, depending on sample characteristics, between 1 and 8 grid openings will likely have to be scanned at high magnification (20,000x) to achieve the target analytical sensitivity for total asbestos structures of $5x10^7$ s/g_{solid}. Similarly, between 15 and 133 grid openings will likely have to be scanned at low magnification (10,000x) to achieve the target analytical sensitivity for long asbestos structures (longer than 5 μm) of $3x10^6$ s/g_{solid}.

The defined sensitivities for total and long structures were easily achieved during the pilot study for the method (Berman et al 1994a).

12.1.2 Precision

Results of the pilot study for this method (Berman et al 1994a) indicate that, when 50 structures are counted, the average relative percent difference observed among eight sets of duplicate samples (four sets of duplicate samples analyzed by each of two laboratories) is 20%.

The precision of an asbestos measurement (in this case, expressed as the relative percent difference) should be inversely proportional to the square root of the number of structures counted. Given that 10 structures are likely to be counted at the target concentrations for this method (see Section 2.1.1) and, based on the precision observed in the pilot study when 50 structures are counted, it is expected that the average relative percent difference achievable for this method should be 43% at the target concentrations listed in Section 2.1.1. Thus, given the analytical sensitivities defined in Section 2.1.1 and the stopping rules defined in Section 11.1, this method is capable of achieving a level of precision that is comparable with the guidelines recommended in the CLP for the analysis of other analytes in soils and defined for this method in Section 2.1.2.

The precision observed during the pilot study for this method is based on specimen grids prepared from the suspension collected in the scrubber of the elutriator (Appendix A). It is expected that the precision achieved for analyses derived from scans of specimen grids derived by direct preparation of filters collected over the isokinetic sampling tube of the dust generator (see Appendix A) will be significantly worse. Procedures are therefore incorporated into this method in which the majority of samples will be analyzed based on preparation of specimen grids from filtered scrubber suspension with a subset of 5 to 10% simultaneously analyzed using specimen grids prepared by a direct transfer technique from filters collected over the isokinetic sampling tube of the elutriator (see Chapter 10). The latter is required to develop a regression relating these two types of preparations so that measurements can be evaluated using existing dose-response factors for asbestos (see Section 2.1.3); existing

dose-response factors tend to be based on the analysis of samples whose preparation corresponds most closely to a direct preparation.

NOTE

It is anticipated that a direct, linear relationship will be observed between asbestos concentrations derived, respectively, from scrubber suspension and from filters collected over the IST opening of the elutriator for samples collected from the same environmental matrix. However, there is little reason to expect that such a relationship will hold across samples collected from different environmental matrices. Therefore, samples to be employed to determine the relationship between asbestos measured from scrubber suspension and from filters mounted over the IST opening of the elutriator should be selected and evaluated separately for each environmental matrix sampled during a study for analysis using this method.

It must be recognized that compromises are required when developing a method of this type. The proposed reporting of samples prepared primarily by an indirect technique with a small subset of directly prepared samples (the latter used primarily to provide a link between the reported measurements and existing dose-response factors) represents such a compromise.

The proposed procedure allows for the determination of *relative* concentrations with maximum precision so that arbitrarily small differences in concentrations can be easily distinguished. Among other things, this will facilitate distinguishing upwind and downwind concentrations by a source. At the same time, somewhat lower precision is considered acceptable for estimating the *absolute* risk associated with any particular measurement.

NOTE

The uncertainty of the proposed regression to link results from directly and indirectly prepared samples is expected to be attributable almost exclusively to the limited precision of the directly prepared samples. Therefore, use of the proposed regression for estimating risks may not significantly increase the uncertainty of such a risk analysis beyond what would be associated with assigning an estimated risk to an asbestos concentration derived from a directly prepared sample in any case.

It is also assumed that a sufficient number of samples (i.e. a minimum of approximately 10) will be co-prepared and analyzed to allow reasonable confidence in the results of the regression.

12.1.3 Accuracy

Based on the results of the pilot study (Berman et al. 1994a), there appears to be a problem with laboratory bias during the counting of asbestos structures in support of this method. The bias is introduced specifically during the analysis of specimen grids because duplicate samples prepared by the same laboratory using identical procedures nonetheless yield significantly different counts by analysts from each of two laboratories.

Given that analysts within a single laboratory appear to be capable of achieving good agreement on counting (also demonstrated in the pilot study), the problem of between-laboratory bias will need to be addressed by the implementation of an aggressive inter-laboratory quality control (QC) program in which samples are regularly shared among laboratories and a procedure for verified counting is instituted. It may also be useful to promote meetings with discussion sessions in which analysts from different laboratories discuss the interpretation of structures viewed simultaneously (either directly from a microscope or from a video).

12.1.4 Asbestos Characteristics

As was observed during the pilot study (Berman et al. 1994a), this method is easily capable of preserving information concerning the distribution of the sizes and shapes of asbestos structures that are likely to be released from environmental matrices that are disturbed by natural or anthropogenic forces. As indicated in Section 2.1.3, such information is critical to evaluating the potential health effects of the asbestos dusts generated by releases from asbestos-containing matrices (see, for example, Berman and Crump 1989).

Interferences and limitations concerning the ability to identify and characterize asbestos structures using the counting and identification rules of the ISO Method, as adopted for this method (Section 11.1), are described in the ISO Method (Chatfield 1993).

12.1.5 Reporting Requirements

As indicated in Section 11.4, the concentrations of asbestos structures (of any defined size or type) that are measured using this method can be easily reported either as a function of the mass of the sampled material or as a function of the mass of respirable dust in the sampled material. This should allow sufficient flexibility to facilitate use of results from this method in concert with any of the fate and transport models that may be employed to predict exposure (Section 2.1.4).

12.2 QUALITY CONTROL REQUIREMENTS

The quality assurance/quality control (QA/QC) requirements indicated in the ISO Method (Chatfield 1993) shall be considered relevant and appropriate when using this method. In addition, the following blank and duplicate/replicate schedule shall be employed when running samples using this method.

12.2.1 Blanks

The following blanks shall be collected routinely in concert with use of this method:

- lot blanks or filter blanks. Two filters from each lot of 50 filters obtained from the manufacturer shall be prepared using a direct transfer procedure and analyzed to assure that background contamination on the filters does not exceed 10s/mm^2 (Section 6.6). Only filters from lots whose blanks pass the defined criterion shall be used in support of this method;

- laboratory blanks. A sufficient number of laboratory blanks shall be collected, prepared using a direct transfer technique and analyzed to show that the room in

which bulk samples are handled and prepared satisfy the requirements defined in Section 10.6 of Chatfield and Berman (1990). When laboratory blanks indicate that room air is out of compliance with the stated criterion, use of this method is to cease until appropriate corrective actions are completed;

- field blanks. Field blanks shall be collected during any sample collection activities performed in association with use of this method. The number of such blanks to be collected and the schedule for their analysis shall be determined based on the complexity of the anticipated sampling scheme and shall be defined as part of the sampling plan for the site. QC criteria for field blanks will also be set as part of the planning for the study;

- equipment blanks. Equipment blanks are collected at the beginning of each run of the dust generator, as described in Section 9.4.3. Equipment blanks do not generally need to be analyzed on a regular basis but shall be stored in case their use is required to help determine the source of contamination that may be discovered by some other means;

- run blanks. Run blanks are also collected at the beginning of each run of the dust generator, as described in Section 9.4.3. One run blank shall be analyzed routinely for the first run completed on any particular sample. The remaining run blanks generated in association with a particular sample shall be stored in case their use is required to help determine the source of contamination that may be discovered by some other means.

  Should asbestos structure counts on run blanks exceed the target criterion of 10 s/mm^2, use of this method shall cease until appropriate corrective actions have been completed and new run blanks are shown to achieve the stated criterion; and

- scrubber blanks. Scrubber blanks shall be derived by collecting a 1 ml aliquot of scrubber liquid (after the scrubber is loaded and assembled for a run but before heating of the scrubber is initiated), diluting the aliquot to 10 ml, and completing the preparation of the diluted aliquot as described in Section 9.4.6. Scrubber blanks do not need to be analyzed routinely but shall be stored in

  case their use is required to help determine the source of contamination that may be discovered by some other means.

In addition to the above listed blanks that must be collected routinely in association with use of this method, the following may prove helpful for identifying the source of any contamination that might be detected in association with use of the dust generator:

- modified run and scrubber blanks. Modified run and scrubber blanks may be generated by setting up and operating a clean dust generator *without* sample. Filters may be collected from either the ME or the IST opening of the dust generator at any point of such a run. Similarly, aliquots of scrubber liquid may be withdrawn from the scrubber at any point. Such blanks may prove useful for determining whether contamination is being introduced by any of the components of an operating dust generator (including, for example, the constant

humidity solution, the rotating tumbler, the elutriator tube, the air transfer lines, and/or the glassware or liquid of the boiling scrubber); and

- post-run scrubber blanks. A post-run scrubber blank may be generated by repeating the rinse of the transfer lines, condensers, and scrubber flask immediately after the quantitative rinse conducted for a particular sample (see Section 9.4.6). The resulting liquid must then be weighed, diluted quantitatively, and prepared in the same manner as described for the scrubber suspension of a sample (Section 9.4.6). The normalized concentration of asbestos structures found in such a blank shall represent no more than 10% of the concentration of asbestos structures observed in the sample prepared immediately prior to collecting the post-run blank. Higher blank concentrations shall be considered unacceptable. If blank concentrations are observed, procedures for quantitative rinsing shall be reviewed, modified, and tested until losses can be shown to be acceptable.

NOTE

Asbestos observed in post-run blanks constitute asbestos that is lost from a sample during preparation.

12.2.2 Duplicates/Replicates

A fixed fraction (5 to 20%) of the samples collected in the field in support of this method shall be collected as spatial duplicates (two samples collected at immediately adjacent locations). These shall be labeled and sent to the laboratory in such a manner so as to assure that laboratory personnel cannot identify them as duplicates. The frequency of collection of spatial duplicates shall be defined as part of the sampling plan for the site. Comparison of the results of the analysis of such samples provides a measure of all of the components of total precision except population variability.

As indicated previously (Section 8.2.1), 100% of the samples shipped from the field are to be shipped as duplicate pairs. The laboratory shall randomly select 10% of the duplicate samples shipped from the field and shall analyze both samples of the pairs so selected. Comparison of the results of the analysis of such samples, which are homogenized splits of the same sample, provides an indication of the precision achieved by sample preparation and analysis.

Should analysis of duplicate pairs indicate an unacceptable degree of variability (i.e. a relative percent difference greater than 50%), replicate counts shall be performed on designated samples by multiple analysts in the laboratory (or by the same analyst on different days). Laboratory management shall assign such counts so as to assure that analysts cannot determine which counts are replicates. Results of such replicate counts shall serve to distinguish whether the major source of variability observed among duplicate pairs is due to analysis or to sample preparation. Appropriate corrective actions may then be devised.

13.0 REPORTING REQUIREMENTS

13.1 FIELD AND LABORATORY NOTEBOOKS

Over the course of the project, information critical to the proper reporting and interpretation of each sample analysis will be developed both in the field and in the laboratory. Formal procedures are required to preserve such information and to allow for the documentation of attendant information that, while not employed directly in the calculation of results, may provide insight into the interpretation of such results.

13.1.1 Field Notebooks

During sample collection, detailed notes will be kept in a standard field or laboratory notebook that is bound and pre-paginated. All entries to the notebook are to be dated and initialed. Information to be recorded during sample collection should include (but is not limited to):

(a) reference to this method;

(b) the project title, identification of the site, and the names and titles of field personnel participating in the sampling effort;

(c) identification of each sample or fraction of each sample handled in the field;

(d) the date and time that each sample was collected and the time interval during which each sample was prepared in the field;

(e) the location from which each sample was collected and the manner in which sample locations were selected (including reference to the sampling plan under which sampling was conducted);

(f) a general description of the physical appearance of the materials sampled;

(g) the types of equipment and the procedures employed to collect each sample;

(h) a summary of the procedure employed for field preparation of each sample (including, for example, the number of passes through a riffle splitter employed for homogenization, and the number of splits required to reduce the sample to the size required);

(i) the relevant identities and weights of each fraction of sample handled during field preparation (including specifically the weights of the coarse and fine fractions separated from each sample during sieving);

(j) other relevant field observations (including, for example, the meteorological conditions under which sampling was conducted); and

(k) the identification, weight, and intended destination of each sample shipped from the field.

13.1.2 Laboratory Notebooks

During sample handling, preparation, and analysis, detailed notes will be kept in a laboratory notebook that is bound and pre-paginated. All entries to the notebook are to be dated and initialed. Information to be recorded during sample preparation and analysis should include (but is not limited to):

(a) reference to this method;

(b) identification of each sample or fraction of each sample received from the field and the date and time that each sample was received;

(c) identification of all laboratory personnel who participate in the preparation and analysis of samples and the specific operations performed by each;

(d) identification of reagents, equipment, and supplies employed during sample handling, preparation, and analysis;

(e) relevant material weights and/or volumes;

(f) the lot number and manufacturer of the filters employed during sample preparation;

(g) a summary of the procedure employed for laboratory preparation of each sample (including, for example, the number of passes through a riffle splitter employed for homogenization and the number of splits required to reduce the sample to the size required);

(h) the relevant identities and weights of each fraction of sample handled during laboratory preparation;

(i) the setup conditions employed for the dust generator (including, for example, the identity of the salt employed for humidity control, air flow conditions, and the setting on the variable voltage transformer employed for the scrubber);

(j) the date and starting time, description (including, primarily, the tumbler rotational speed), and sample identification for any run conducted on the dust generator;

(k) the weight and identity of the sub-sample placed in the dust generator;

(l) the identities, time intervals of collection, the starting and ending weights, and the net weights of filters collected during a run on the dust generator that are to be used to determine the rate of dust generation;

(m) the figures and calculations employed to determine the rate of dust generation and the quantity of dust in the sample;

(n) the identity, time interval of collection, starting and ending weights, and the net weights of filters collected during a run on the dust generator that are to be used to generate specimen grids for asbestos analysis;

(o) the identity, time interval of collection, the weight/volume, dilution factor, aliquot identification, and filter identification for the collection, handling, and filtering of scrubber suspension;

(p) the identity of all filter sections used for preparation of specimen grids and the identity of the specimen grids (include a description of the sector and radial distance from the center of the filter represented by each section);

(q) energy levels and settings for instruments employed for analysis;

(r) flow rates, pressures, temperatures, and other relevant physical parameters that potentially impact procedures;

(s) room conditions (i.e. temperature, relative humidity, and ventilation rates) prevailing during sample preparation;

(t) other relevant observations (including, for example, any difficulties encountered during preparation and analysis and any procedural changes incorporated into the method that are necessitated by such difficulties);

(u) documentation of all calculations performed in support of preparation and analysis;

(v) all relevant QA/QC measurements (including the results of the analysis of all blanks, duplicates, and replicates--see Chapter 12); and

(w) a detailed time log of events including the time that particular procedures are initiated and completed for each sample.

13.2 FIELD ACTIVITIES REPORT

To assure that the field information required to complete estimation of dust and asbestos concentrations and release rates are provided to the data users, a field activities report must be completed and must be submitted to the laboratory along with the corresponding samples. Laboratory personnel are then to attach this report directly to their batch report, which shall cover the corresponding batch of samples.

The field activities report shall include the following for each sample batch, at a minimum:

(a) the project title and the identification of the site;

(b) reference to this method;

(c) reference to the sampling and analysis plan under which samples were collected;

(d) a brief description of the objectives for sampling;

(e) a brief description of the procedures employed for selecting sampling locations and the motivation for employing such procedures;

(f) for each sample in the batch accompanying the report:

- the identifier for each sample fraction submitted to the laboratory;
- the identifier for the original sample from which each submitted sample fraction was derived;
- the type of equipment and reference to (or a brief description of) the procedures employed for collection of the original sample;
- the coordinates at which each original sample was collected[36];
- the total mass of the original sample from which each sample fraction originated and the masses of the coarse and fine fractions separated during sieving of the sample in the field;
- a brief description of the procedures employed for sample homogenization and for sample splitting;
- the date and time that each sample was collected and the date and time that each sample fraction was prepared in the field; and
- a brief discussion of any deviation from the sampling and analysis plan not covered in e.

An example of the format to be employed for a field activities report is provided in Figure 13-1.

13.3 SAMPLE ANALYSIS REPORT

The sample analysis report for each sample shall include the following at a minimum:

(a) reference to this method;

(b) reference to the sample identification and batch number for the sample;

(c) the date and site from which the sample was collected;

(d) the weights and identities of the coarse and fine fractions of the sample and the sub-sample of the fine fraction sent for analysis;

(e) the weights and identities of any splits or other fractions of the sample generated during laboratory preparation;

(f) the weight and identity of the sub-sample placed in the dust generator;

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[36] If the submitted sample fraction is a sub-sample of a composite, what should be described here is the specific area of concern (or portion thereof) that is intended to be represented by the composite),

FIGURE 13–1
FORMAT FOR THE FIELD ACTIVITIES REPORT

Name of field activities contractor
Address of contractor
Contact Name
Telephone Number

Report Title:
Report Number:

Date

PROJECT/SITE:
Name
Address

METHODS AND PROCEDURES:
Field Investigation Design: *(Reference the sampling plan)*
Sample Collection and Handling: *(Reference this method)*

SAMPLING OBJECTIVES:
(Complete a brief description here)

SAMPLING LOCATION SELECTION PROCEDURE:
(Complete a brief description here)

SAMPLE DATA

Field Sample Number	Location Identifiers	Mass of Sample	Mass Fine Fraction	Mass Coarse Fraction	Mass Sample Split	Mass Duplicate Split	Sample Split ID	Duplicate Split ID	Date Sampled	Time Sampled	Comments: *(Include identification of all field sampling and field preparation procedures employed)*

An example of the format to be employed for a sample analysis report is presented in Figure 13-2.

13.4 SAMPLE BATCH REPORTS

In addition to the sample analysis report for each sample, provide a summary page for each batch of samples representing an entire project. The summary sheet shall include:

(a) the project title;

(b) reference to this method;

(c) the date that samples were collected, the date they were received by the laboratory, and the date they were analyzed;

(d) a summary listing of sample results including:

- the sample number;
- the estimated concentration of respirable dust in the sample;
- the analytical sensitivity achieved for each size/type category of interest;
- the total number of structures of each size/type category of interest counted;
- the concentration of asbestos structures of each size/type category of interest in the sample and in the respirable dust of the sample (both reported along with corresponding 95% confidence limits); and
- the concentration of asbestos structures of each size/type category of interest estimated for the environmental matrix that was sampled in the field (reported along with corresponding 95% confidence limits).

An example of the format to be employed for sample batch reports is presented in Figure 13-3.

FIGURE 13–2
SAMPLE ANALYSIS REPORT FORMAT

Laboratory Name
Laboratory Address
Laboratory Contact
Telephone Number

Report Date
Project Name *(Optional)*

METHODS:
(reference this method)

Date Analysis Started (M/D/Yr)
Date Analysis Completed (M/D/Yr)
Analyst(s) Initials

Laboratory Sample No.
Field Sub–Sample Identification No.
Field Preparation Technique **(Attach a Copy of the Relevant Field Activities Report)**
Additional Laboratory Preparation Procedures *(describe any employed)*
Sample Drying
Sample Splitting
Other

TEM Analysis:
Effective Area of Analytical Filter (sq mm)
(Indicate whether from Scrubber or from IST Opening)
Magnification
Grid Opening Area (sq mm)
Number of G.O. Scanned
Asbestos Structure Size and Type Categories of Interest *(see Chatfield 1993)*
Minimum Acceptable Structure Identification Category *(see Chatfield 1993)*

Dust Generator
Mass of Sample Tumbled (g)
Air Flow Rate Through ME Opening of Dust Generator (ml/min)
Air Flow Rate Through IST Opening of Dust Generator (ml/min)
Air Flow Rate Through Scrubber (ml/min)
Estimated Total Air Flow Rate Through Elutriator (ml/min)

Total Mass of Dust Collected on Dust Filters (g)
Time of Dust Collection *(24 hour clock)* at 30 rpm
Start:
Stop:
Time of Dust Collection *(24 hour clock)* at 60 rpm
Start:
Stop:
Estimated first–order rate constants for dust generation (min^{-1})
At 30 rpm:
At 60 rpm:
Estimated Starting Mass of Respirable Dust in Sample (g)
(Attach time plots and calculations)

Samples from the Scrubber Suspension
Total Volume of Scrubber Suspension (ml)
Estimated Mass of Dust Collected in Scrubber Suspension (g)
Volume of Aliquot Withdrawn from Scrubber Suspension (ml)
Volume into which Scrubber Aliquot Diluted (ml)
Dilution Factor (dimensionless)
Volume of Aliquot Filtered *(from Diluted Suspension)* (ml)

Samples from the Isokinetic Sampling Tube (IST) Opening of the Dust Generator
(indicate whether 30 or 60 rpm run)
Time of Collection *(24 hour clock)*
Start:
Stop:
Estimated Mass of Dust Collected on Filter

FIGURE 13–2
SAMPLE ANALYSIS REPORT FORMAT (Cont.)

Laboratory Name

Report Date

Laboratory Sample No.

	Low Magnification	High Magnification
Chrysotile Asbestos Analysis Results:		
No. of Total Chrysotile Asbestos Structures	XXX	
No. of Long (> 5 um) Chysotile Asbestos Structures		
No. of Total Chrysotile Asbestos Fibers/Bundles	XXX	
No. of Long (> 5 um) Chysotile Asbestos Fibers/Bundles		

	Low Magnification	High Magnification
Amphibole Asbestos Analysis Results:		
No. of Total Amphibole Asbestos Structures	XXX	
No. of Long (> 5 um) Amphibole Asbestos Structures		
No. of Total Amphibole Asbestos Fibers/Bundles	XXX	
No. of Long (> 5 um) Amphibole Asbestos Fibers/Bundles		

(Indicate Amphibole Mineral Type)

ESTIMATED CONCENTRATIONS OF RELEASABLE ASBESTOS IN SAMPLE

	Conc.	95%UCL
Total Chrysotile Structures per g Sample:		
Total Amphibole Structures per g Sample:		
Total Asbestos Structures per g Sample:		
Long Chrysotile Structures per g Sample:		
Long Amphibole Structures per g Sample:		
Long Asbestos Structures per g Sample:		

Estimated Analytical Sensitivity: *(structures/g sample)*

ESTIMATED CONCENTRATIONS OF RELEASABLE ASBESTOS IN RESPIRABLE DUST OF SAMPLE

	Conc.	95%UCL
Total Chrysotile Structures per g Dust:		
Total Amphibole Structures per g Dust:		
Total Asbestos Structures per g Dust:		
Long Chrysotile Structures per g Dust:		
Long Amphibole Structures per g Dust:		
Long Asbestos Structures per g Dust:		

Estimated Analytical Sensitivity: *(structures/g dust)*

(Attach a Copy of the TEM Raw Data Sheets)

FIGURE 13–3
SAMPLE BATCH REPORT FORMAT

Laboratory Name
Laboratory Address
Laboratory Contact
Telephone Number

Report Date
Project Name *(Optional)*

METHODS:
(reference this method)

RELEASABLE ASBESTOS IN RESPIRABLE DUST

Laboratory Sample I.D.	Respirable Dust Conc (g/g smpl)	Total Asbestos Analytical Sensitivity (s/g dust)	Total Asbestos Conc (s/g dust)	Total Asbestos 95% UCL (s/g dust)	Long Asbestos Analytical Sensitivity (s/g dust)	Long Asbestos Conc (s/g dust)	Long Asbestos 95% UCL (s/g dust)	Dust Generation Rate (at 30 rpm) (min^{-1})	Dust Generation Rate (at 60 rpm) (min^{-1})	Comments:

RELEASABLE ASBESTOS IN LABORATORY SAMPLES

Laboratory Sample I.D.	Respirable Dust Conc (g/g smpl)	Total Asbestos Analytical Sensitivity (s/g smpl)	Total Asbestos Conc (s/g smpl)	Total Asbestos 95% UCL (s/g smpl)	Long Asbestos Analytical Sensitivity (s/g smpl)	Long Asbestos Conc (s/g smpl)	Long Asbestos 95% UCL (s/g smpl)	Comments:

RELEASABLE ASBESTOS IN FIELD SAMPLE MATRICES

Laboratory Sample I.D.	Field Sample I.D.	Mass Fine Fraction (g)	Mass Coarse Fraction (g)	Adjusted Respirable Dust Conc (g/g mtrx)	Adjusted Total Asbestos Conc (s/g mtrx)	Adjusted Total Asbestos 95% UCL (s/g mtrx)	Adjusted Long Asbestos Conc (s/g mtrx)	Adjusted Long Asbestos 95% UCL (s/g mtrx)	Comments:

14.0 REFERENCES

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Chatfield, E.J. "Ambient Air: Determination of Asbestos Fibres, Indirect-Transfer Transmission Electron Microscopy Procedure." Submitted to: ISO/TC 146/SC 3. 1993.

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APPENDIX A:

CONSTRUCTION AND OPERATION OF A DUST GENERATOR

APPENDIX A:
CONSTRUCTION AND OPERATION OF A DUST GENERATOR FOR ISOLATING THE RESPIRABLE FRACTION OF MATERIAL FROM SOILS OR OTHER BULK SAMPLES

The dust generator incorporated into this method for the determination of asbestos in soils and bulk materials is designed to isolate the respirable fraction of material (including the releasable fraction of respirable asbestos) that is present within the matrix of the parent sample. A description of the apparatus is provided below along with a brief discussion of the theory of its operation. Figures depicting design of the prototype are also provided in the last section of this appendix to facilitate design and construction of similar equipment.

A.1 DUST GENERATOR DESCRIPTION

The dust generator is composed of a tumbler, a vertical elutriator, a dust collection system, and a scrubber. A schematic diagram of the apparatus is presented in Figure A-1 and a photograph of the apparatus is shown in Figure A-2. The appurtenant equipment required to operate the dust generator is also shown in Figure A-2. This includes:

- a DC motor to drive the tumbler;
- a constant humidity chamber to control the humidity of the air that flows into the dust generator. This is the clear plastic box enclosing the tumbler in the photograph;
- the pumps required to create an air flow through the dust generator and the flow controllers required to monitor and apportion air flow through the various filters and the scrubber; and
- the heating mantle, variable voltage transformer, and cooling towers required for the scrubber.

A.1.1 The Tumbler

The tumbler is located in the clear plastic enclosure at the bottom of the dust generator (Figures A-2 and A-3). It is a long shallow tube of square cross section that is approximately 1 and 1/8 inches in height and width and has an overall length of 10 inches (Figure A-4).

The tumbler is driven by a variable speed DC motor, which rotates the tumbler around its long axis. The tumbler's rotation rate can be varied over the range of 10 to 150 revolutions per minute (rpm). The DC motor is attached to the tumbler using a slip-on type flexible coupling so that they can be readily detached simply by pulling the motor away from the tumbler. The tumbler can also be readily detached from the connection tube to the elutriator; they are connected with a slip fit over the outer race of the ball bearing assembly that is welded to the end of the tumbler (Figure A-4).

FIGURE A-1

SCHEMATIC OF DUST GENERATOR

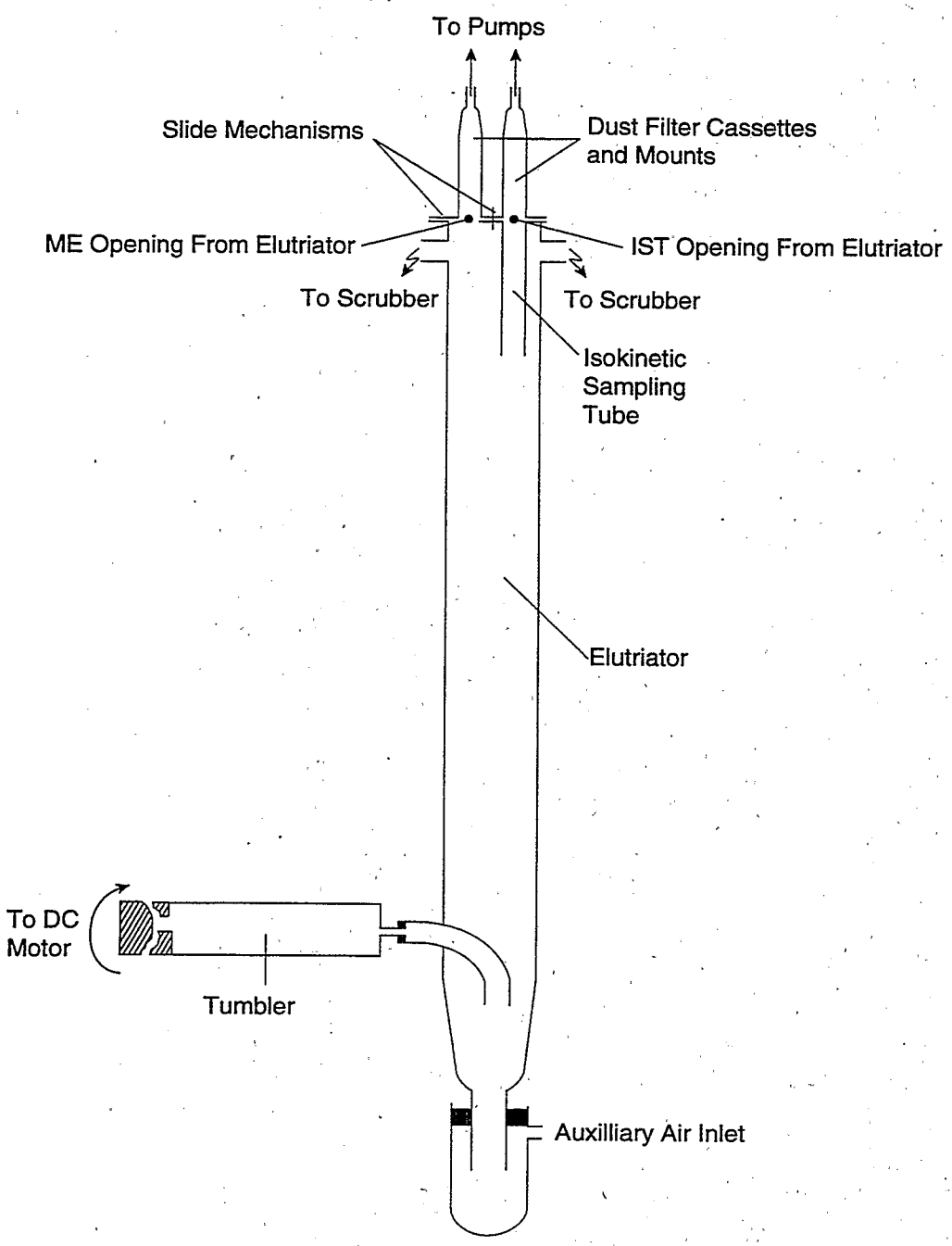


FIGURE A-2

THE DUST GENERATOR

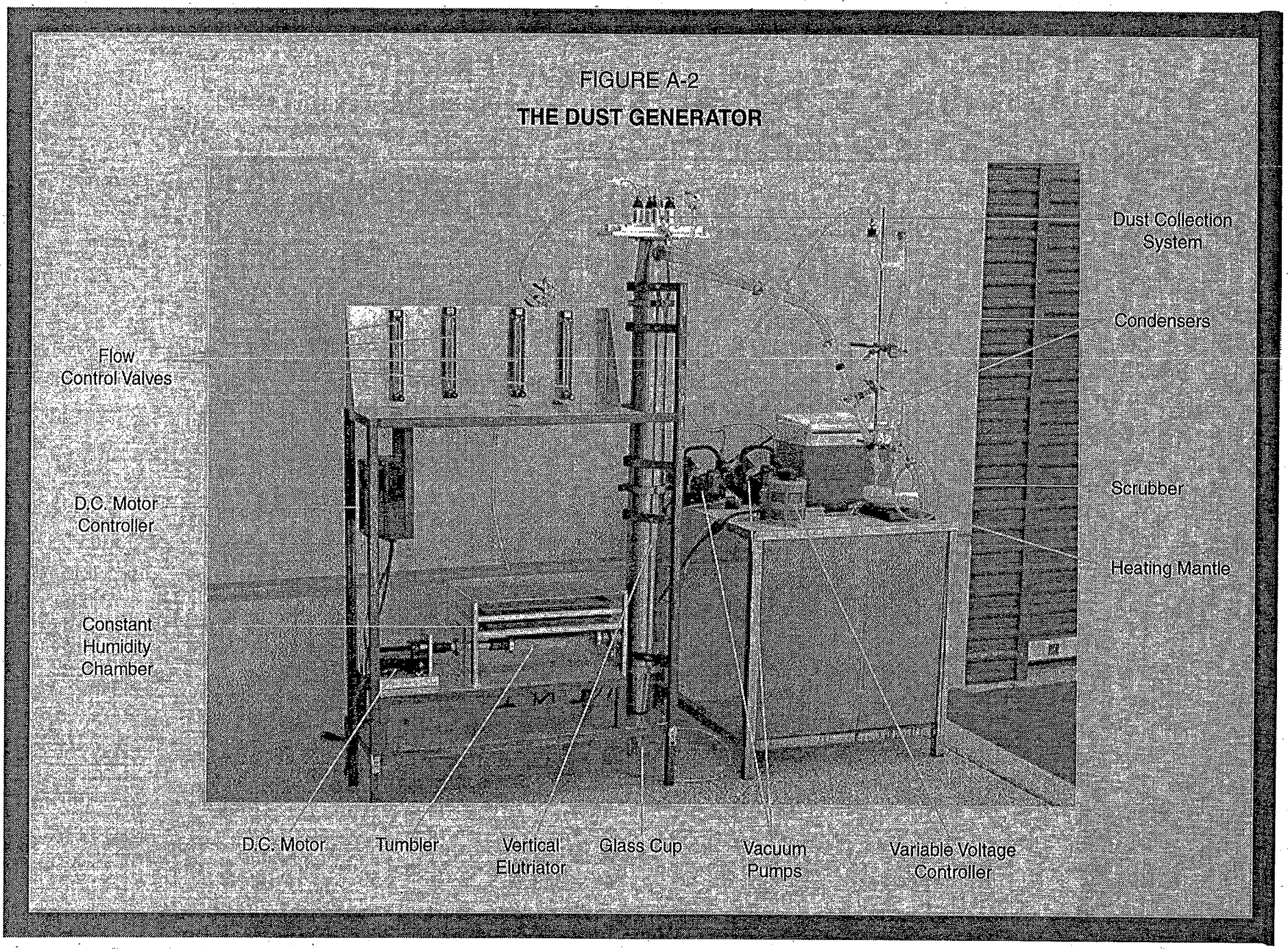


FIGURE A-3

TUMBLER IN CONSTANT HUMIDITY CHAMBER

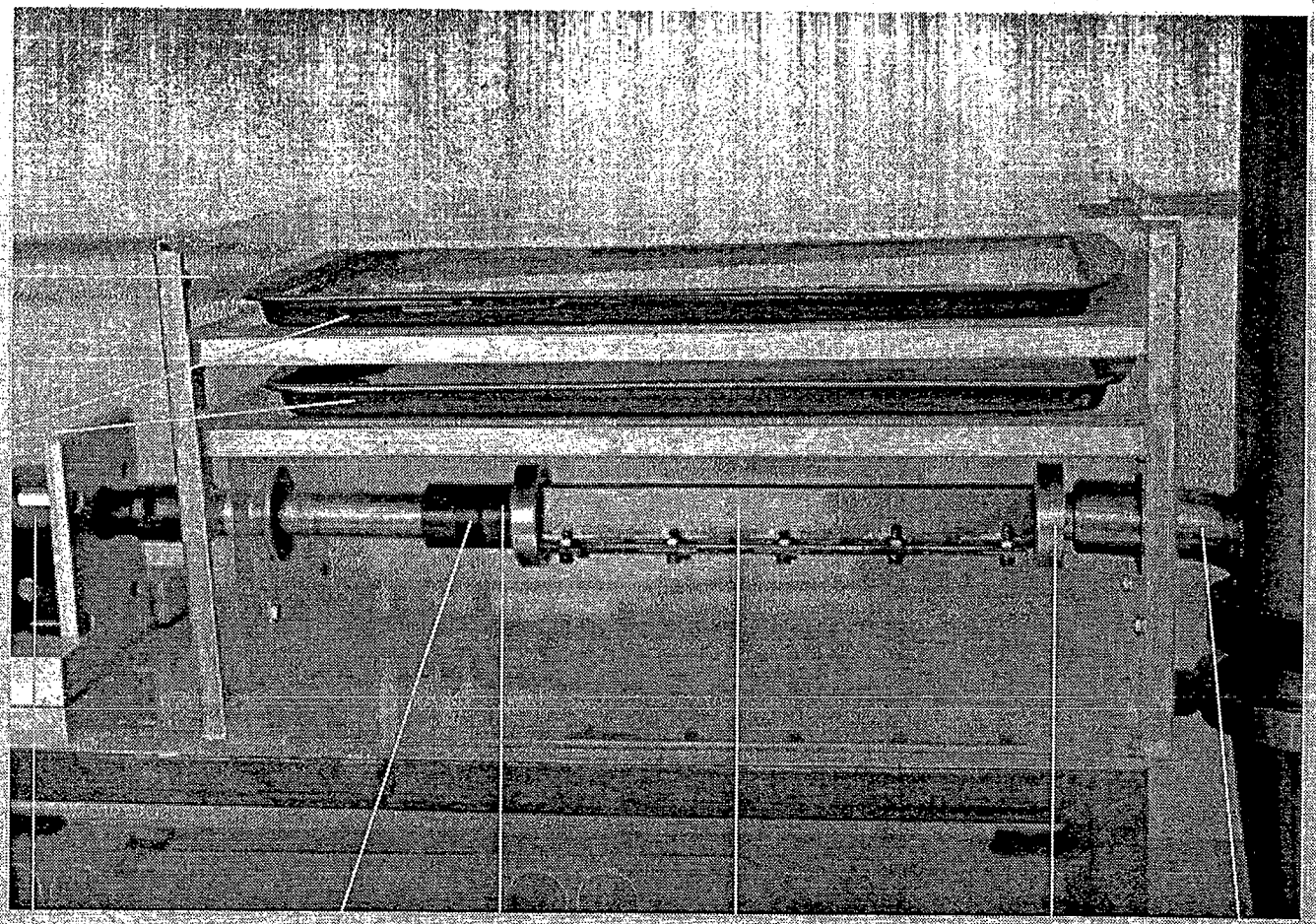
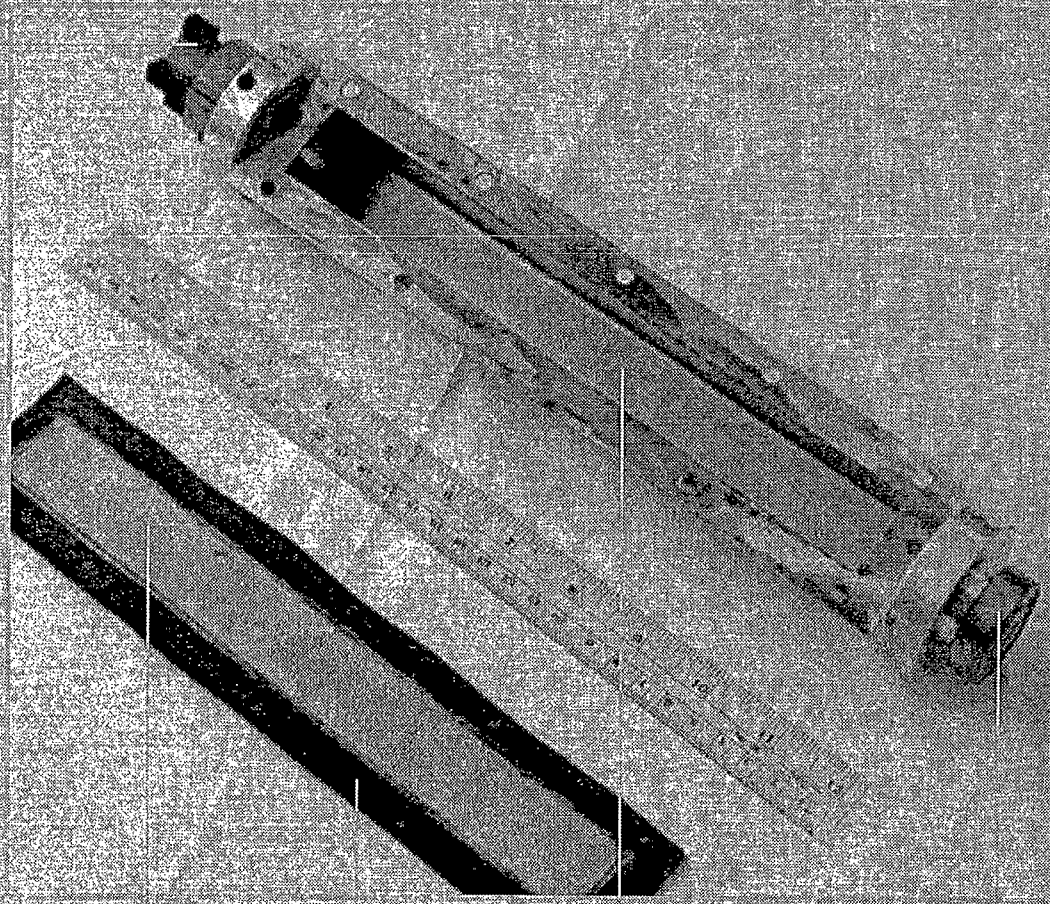


FIGURE A-4

TUMBLER ASSEMBLY



NOTE

The seal between the tumbler and elutriator need not be air tight. In fact, a *small* leak at this fitting will reduce the chance that particles discharged from the tumbler will settle in the entrance tube to the elutriator. However, such a leak must not be more than 10% of the total airflow through the tumbler.

Once the tumbler has been detached from the motor and elutriator, it can simply be lifted out of the constant humidity chamber.

The top cover of the tumbler is secured with 10 screws and can be removed so that the tumbler can be loaded or cleaned (Figure A-4). A rubber gasket is employed to assure an airtight seal between the tumbler cover and body.

A.1.2 The Constant Humidity Chamber

The plastic enclosure that houses the tumbler and isolates it from outside air, except for an opening at the top front of the enclosure, is designed as a constant humidity chamber (Figures A-2 and A-3). Air drawn into the front of the enclosure flows successively over two trays (located on shelves at the top of the enclosure) before being drawn into the tumbler. The trays are designed to hold a saturated salt solution that is selected to exhibit a vapor pressure equal to the relative humidity desired for running the dust generator. The path length over the two trays is designed to assure that air drawn through the device will come to equilibrium at a relative humidity within a few percent of the desired value (assuming that the outside air exhibits a starting humidity within the range common to Pasadena, California[1]); the path length must be sufficient to allow adequate exchange of vapor between the salt solution and the moving air.

For most studies, a relative humidity of approximately 50% should be employed because this is the humidity at which emission of asbestos is expected to be maximum (Zimon, A.D. 1982). At higher humidities, air moisture tends to wet surfaces so that aggregation decreases particle release. At lower humidities, electrostatic effects tend also to cause aggregation and, therefore, also to decrease emissions. A saturated solution of potassium carbonate dihydrate is recommended for most applications because such a solution maintains equilibrium with air at 43% relative humidity at room temperature (20^{o} C) with less than 1% change in humidity over a range of temperatures varying by several degrees on either side of room temperature.

For special applications of this method, where dust generation is to be run at a different relative humidity than that produced over a saturated solution of potassium carbonate dihydrate, the conditioning pans in the dust generator may be filled with a saturated solution of another salt. Such salts may be selected to yield any of a broad range of conditions. For example, the International Critical Tables provides a list of saturated salt solutions that maintain equilibrium with a broad range of relative humidities in air.

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[1] The prototype device was constructed and tested in Pasadena. The design of the constant humidity chamber may have to be modified slightly (e.g. by increasing the number of trays) for operation in other locales.

A.1.3 The Vertical Elutriator

The vertical elutriator is the tall metal cylinder visible in Figure A-2. Its dimensions were selected to assure that the path length traversed by particles in the elutriator is at least 10 times the diameter of the elutriator (in the direction perpendicular to the air flow). Thus, channeling is expected to be minimized (Dennis, R 1976).

The elutriator separates respirable from non-respirable particles and respirable particles are passed on to dust collection filters and the scrubber. Air flow within the elutriator is adjusted to a velocity such that only those particles in the respirable size range will be lifted to the top. The velocity of air selected for the elutriator determines the rate of air flow through all of the other parts of the dust generator.

The main body of the elutriator is composed of two, 20 in. sections of 4-in. i.d. stainless steel tubing with a tapered, o-ring flange welded to each end of each tube (Figure A-5a and b). These flanges allow the two halves of the main body to be connected to each other and to the top and bottom assemblies of the elutriator using quick-release, pressure clamps (Figure A-5a).

The entrance tube to the elutriator (from the tumbler) is pointed downward at its exit point in the bottom of the elutriator (Figures A-1 and A-5b). This geometry was selected to prevent introduction of a large concentration of particles into the bottom center of the elutriator. This geometry should also promote a flatter velocity profile in the air traveling up the elutriator. It is expected that respirable particles will make the upward turn because the taper at the bottom of the elutriator promotes a higher velocity profile at this location than in the main body of the elutriator (where the air velocity is just sufficient to lift particles in the respirable size range but no larger).

The bottom assembly of the elutriator has also been designed to allow the removal of coarse particles while assuring that respirable particles remain in the elutriator air stream (Figure A-6a and b). This is to minimize buildup of a zone at the bottom of the elutriator of a high concentration of coarse particles through which respirable particles would have to pass before moving up the elutriator to the dust collection system. Thus, it is expected that collisions between respirable particles and coarse particles (with subsequent loss of respirable particles due to aggregation) are minimized.

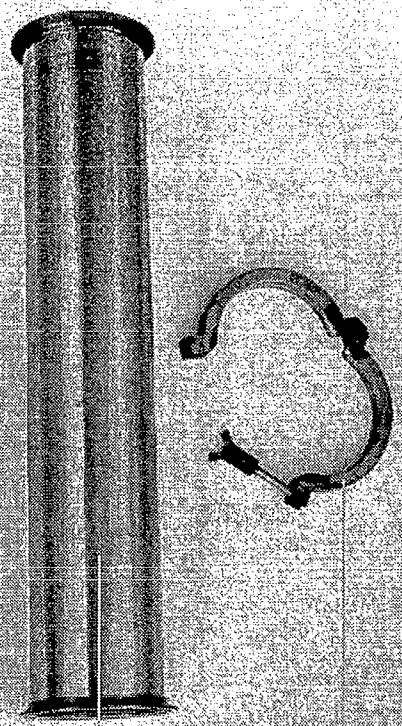
An opening at the bottom of the elutriator allows coarse particles to pass into a removable, glass cup (Figures A-1 and A-6a and b). The glass cup has a small side arm that is connected to a two ft. piece of one quarter in. i.d. Tygon tubing. The far end of the Tygon tube is open to the air. It has been found that, as long the Tygon tube is not crimped or otherwise blocked, air drawn through this tube is just sufficient to prevent respirable particles from passing through the bottom opening of the dust generator into the glass cup while allowing larger particles to do so.

There are four openings where air may exit from the top assembly of the elutriator (Figures A-1 and A-7a and b). The two side openings lead into the scrubber (described below). The two top openings lead into the air filter cassettes of the dust collection system through two different paths; one draws air isokinetically from the uniform portion of the elutriator through a long, thin-walled tube and the other draws air from the top, tapered part of the elutriator (Figures A-1 and A-7a and b). For ease of reference, the opening in the elutriator that draws

FIGURE A-5

MAIN BODY OF VERTRICAL ELUTRIATOR

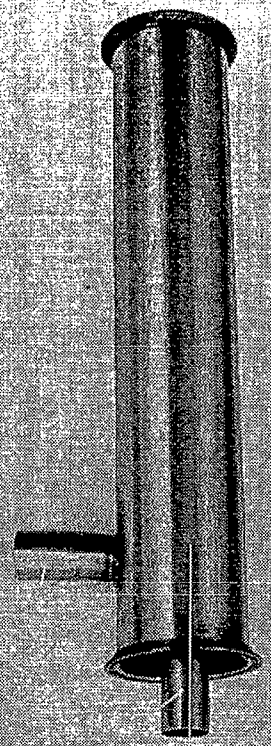
a. Top Section



Body Tube

Quick Release
Pressure Clamp

b. Bottom Section



Entrance Tube
to Elutriator

Body Tube

FIGURE A-6

BOTTOM ASSEMBLY OF VERTICAL ELUTRIATOR

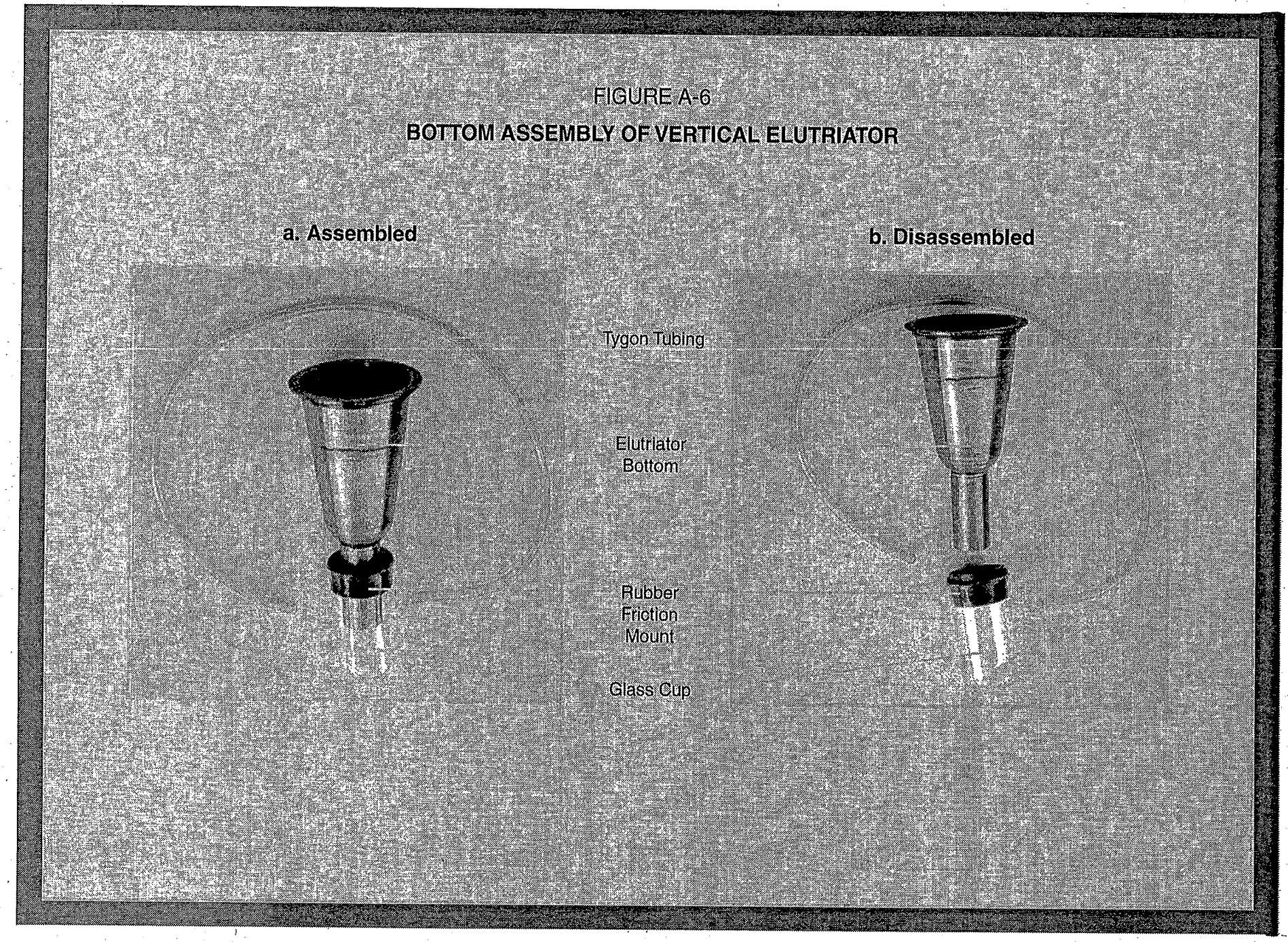
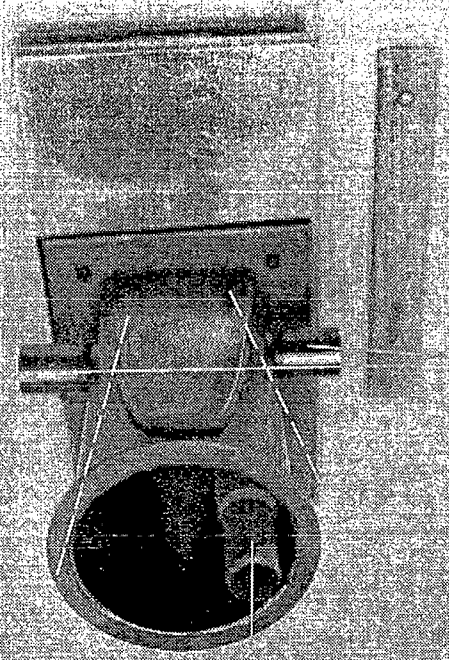


FIGURE A-7

TOP ASSEMBLY OF VERTICAL ELUTRIATOR

a. Underside View



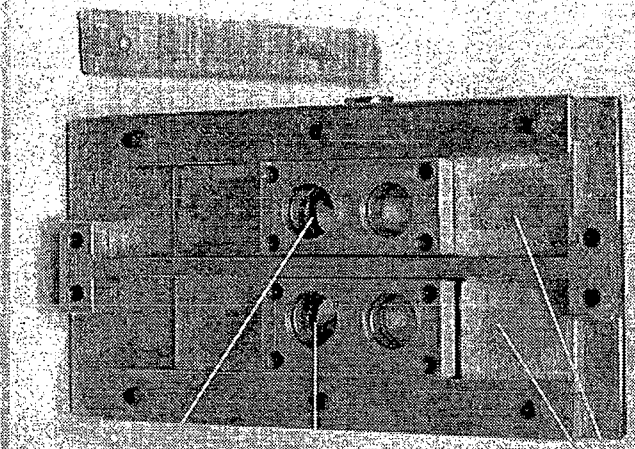
Side Exits
(To Scrubber)

ME Opening

Isokinetic
Sampling Tube

IST Opening

b. Top View



IST Opening to
Elutriator

ME Opening to
Elutriator

Slide Mechanisms

air through the isokinetic sampling tube is labeled the "IST" opening in this document. The opening that draws air from the top of the main body of the elutriator is labeled the "ME" opening. The dust collection system mounted over both of the top openings of the elutriator is described below.

A.1.4 The Dust Collection System

The dust collection system consists of two mounts for filter cassettes welded to each of two sliding mechanisms, which in turn are mounted directly over the exit openings of the top assembly of the vertical elutriator (Figure A-8a and b). An underside view of the tapered top piece of the elutriator was also shown in Figure A-7a in which the isokinetic sampling tube is visible.

The sliding mechanisms permit the filter cassettes aligned over the ME and the IST openings of the elutriator, respectively, to be changed with minimal disturbance of the air flow; at either of the two extreme positions in the travel of each slide mechanism, one of the two cassette mounts are aligned over the corresponding exit opening of the elutriator and the other is effectively isolated from the air flow. Details of the sliding mechanisms and filter mounts are shown in Figures A-7b and A-8a and b.

NOTE

The slides are sealed against the elutriator with o-rings. These must be inspected periodically for wear and replaced if worn.

The design of the filter mounts in the dust collection system proved to be important to the performance of the dust generator; it is critical that these mounts be leak tight. The design that was ultimately adopted is depicted in Figure A-9a and b. The filter mounts each consist of a tapered aluminum base that is glued to the bottom half of a commercially available filter cassette.

The aluminum base of a filter mount is sealed in each of the openings of the slide mechanisms with the o-rings that are visible in Figure A-7b. The bottom half of the plastic, 25 mm filter cassette is glued into the inside taper of the aluminum base. Filters are mounted in the traditional manner between the top and bottom halves of the plastic filter cassette, which are then pressure sealed. To further assure a good seal, pressure tape is also applied to the outside of each filter cassette at the seam where the filters are mounted.

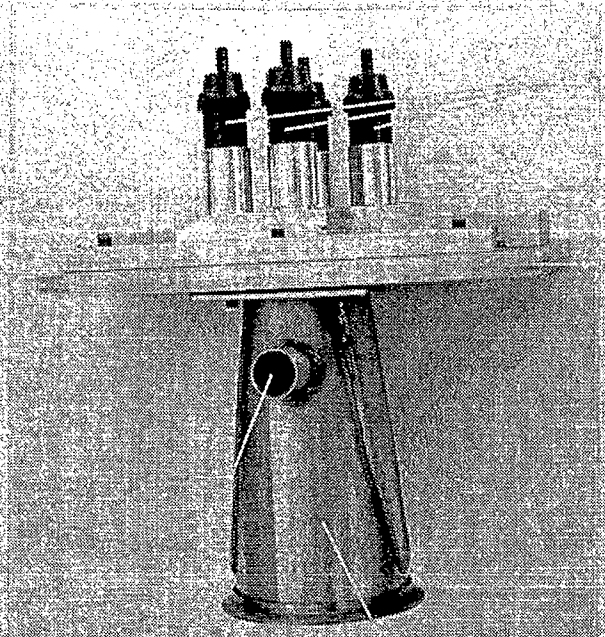
A.1.5 The Scrubber

The scrubber, which can be seen sitting on the table to the right of the main body of the dust generator in Figure A-2, is constructed from ordinary laboratory glassware. In the scrubber, water is boiled in the bottom of a 1 liter, round bottom flask (shown seated within a heating mantle). A straight-jacketed, cold-water condenser is incorporated along the entrance line to the scrubber and a spiral condenser is incorporated along the exit line from the scrubber. An immersion pump circulates water between a cooler containing a water-ice mix and the condensers. Water flowing into the condensers is maintained at approximately 0^{o} C to prevent moisture from leaving the scrubber.

FIGURE A-8

DUST COLLECTION SYSTEM

a. Side View



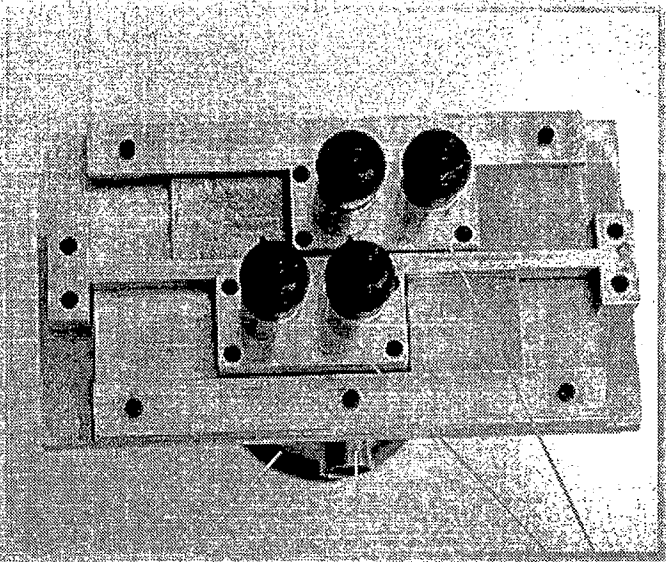
Side Exit Opening of Elutriator (To Scrubber)

Top Assembly of Vertical Elutriator

b. Top View

Filter Mounts

Slide Mechanisms



Top Assembly of Vertical Elutriator

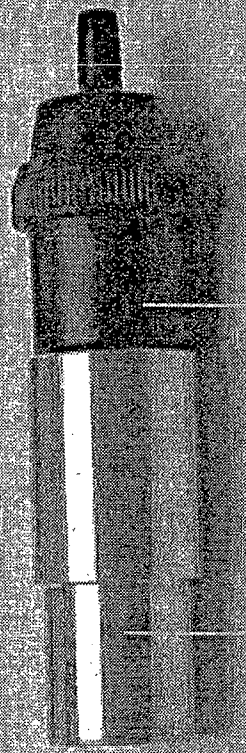
Side Exit Opening of Elutriator (To Scrubber)

Slide Mechanisms

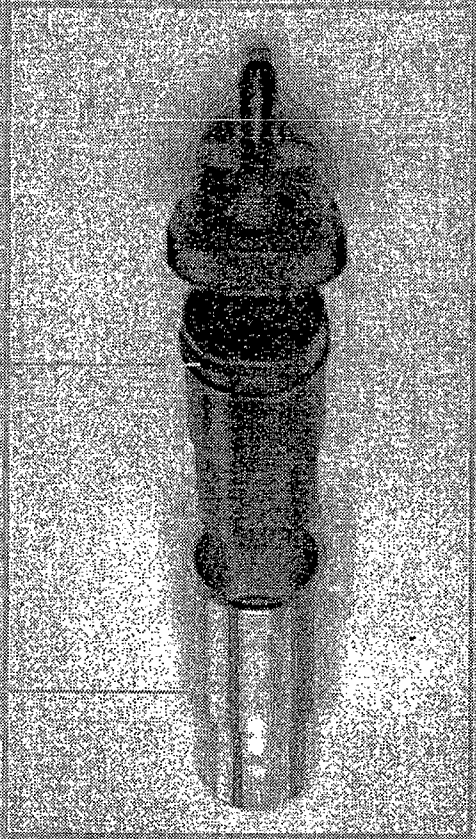
FIGURE A-9

FILTER MOUNTS

a. Assembled Filter Mount



b. Disassembled Filter Mount



Particles in the air stream entering the scrubber serve as nucleation centers around which the steam in the scrubber condenses. The resulting water droplets eventually fall back into the water reservoir at the bottom of the scrubber so that the trapped particles are collected in this reservoir.

A flowmeter and a filter cassette are also placed in the air flow line that exits the scrubber (between the scrubber and the vacuum pump). The primary reason for including the filter cassette is to make the pressure drop through this path approximately equal to the pressure drop through the other filter cassettes mounted on top of the elutriator (the dust collection system). The pressure drop through the elutriator and scrubber is small enough not to affect the flowmeter operation.

A.1.6 The Configuration of Air Transfer Lines

A schematic indicating the configuration of air transfer lines leading to and from the scrubber is presented in Figure A-10. As indicated in the figure, the two side exits from the elutriator are each attached to one ft sections of 1.00 in. i.d. Tygon tubing, which then join at a glass "Y" connector. The stem end of the "Y" connector is attached (with a one ft section of 1.00 in. i.d. Tygon tubing) to a diameter reducing piece of glass that feeds into the entrance condenser to the scrubber through a rubber stopper. The large tubing is held in place at connections by ring clamps (Figure A-2).

Also as indicated in Figures A-10 and A-2, the exit condenser of the scrubber is connected to a flow control valve with 0.25 in. i.d. Tygon tubing. The exit side of the flow control valve is connected first to a 25 mm filter cassette and then to a vacuum pump. Both of these connections also use 0.25 in. i.d. Tygon tubing.

A schematic indicating the configuration of air transfer lines leading from the filter cassette mounts of the dust collection system is depicted in Figure A-11. A photograph of the transfer line connections is also presented in Figure A-12. As indicated in the figures, a 0.25 in. i.d. Tygon line leads from the exit side of each filter cassette to a plastic, stop cock valve. Another Tygon line then leads from each valve to one of two plastic "T" connectors so that the pair of filter cassettes mounted on the slide mechanism over the IST opening of the elutriator are joined (beyond the stop cock valves) and the pair of filter cassettes mounted on the slide mechanism over the ME opening are also joined (beyond the stop cock valves). The common line from the exit side of *each* "T" connector is then connected first to a flowcontrol valve and then to a vacuum pump. All such connections use 0.025 in. i.d. Tygon tubing.

NOTE

This is the configuration of air transfer lines that is appropriate during the operation of the dust generator. The configuration that is appropriate during calibration of air flow is discussed in Section 9.3.4 of the main text.

As indicated previously, the side arm on the bottom cup of the elutriator is connected to a two ft section of 0.25 in. i.d. Tygon tubing that is simply allowed to hang free (Figure A-6a).

FIGURE A-10

TUBING CONNECTIONS FOR THE SCRUBBER OF THE DUST GENERATOR

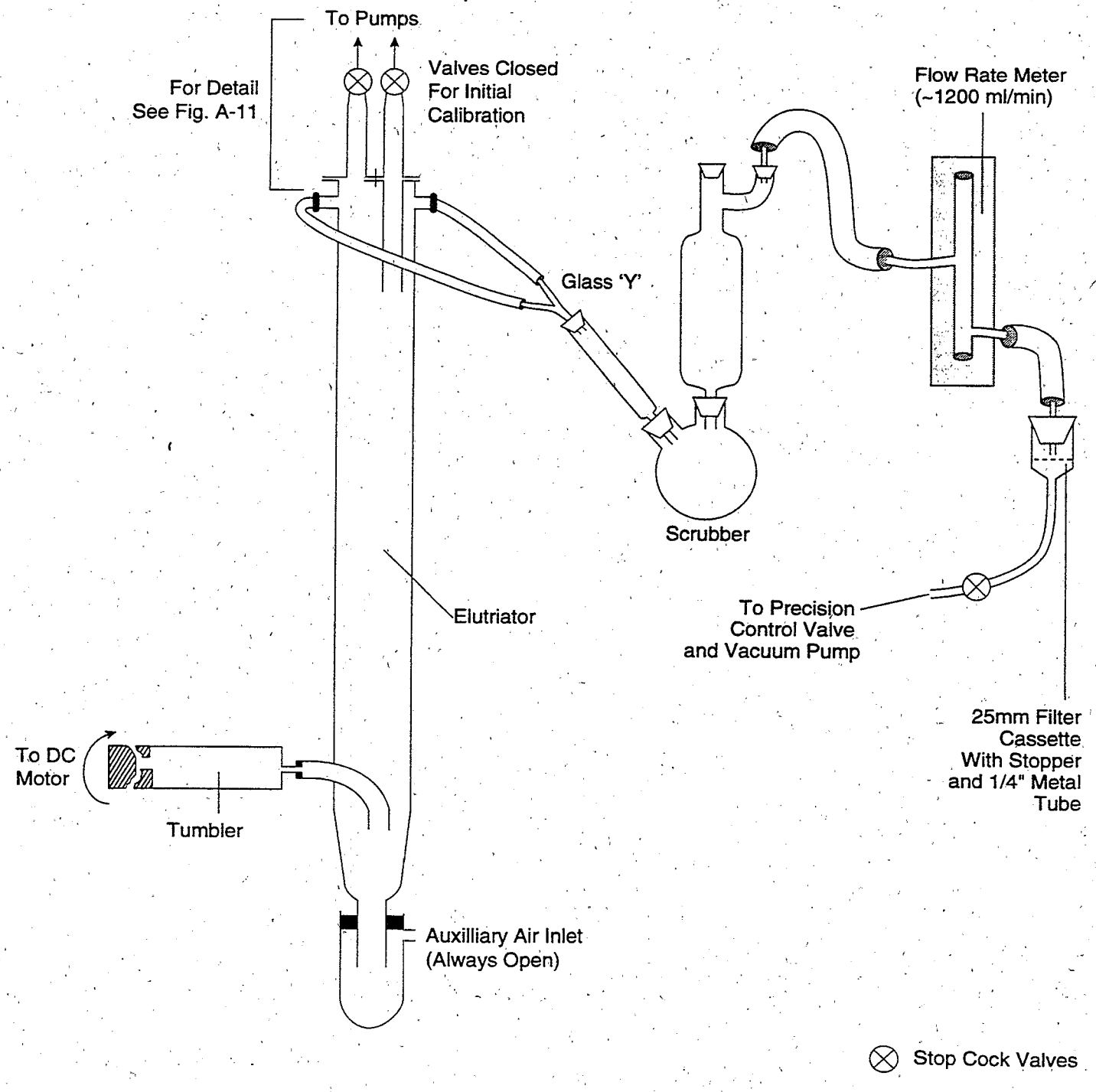
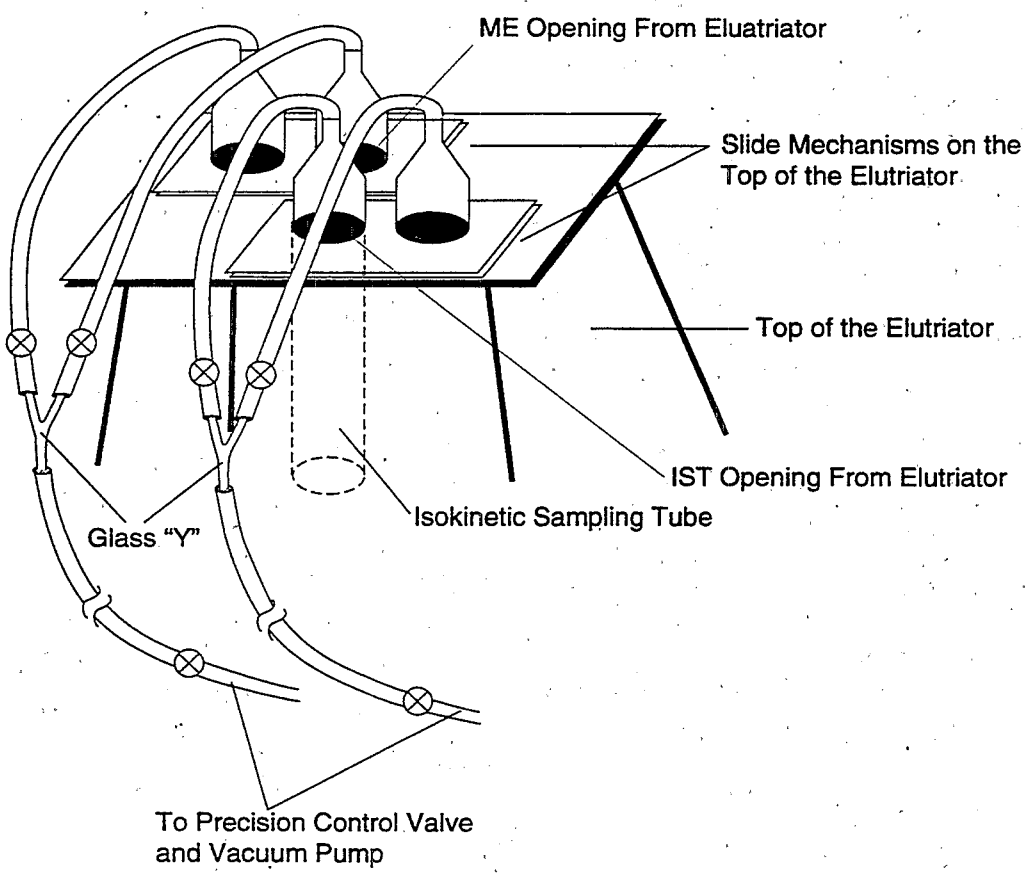


FIGURE A-11

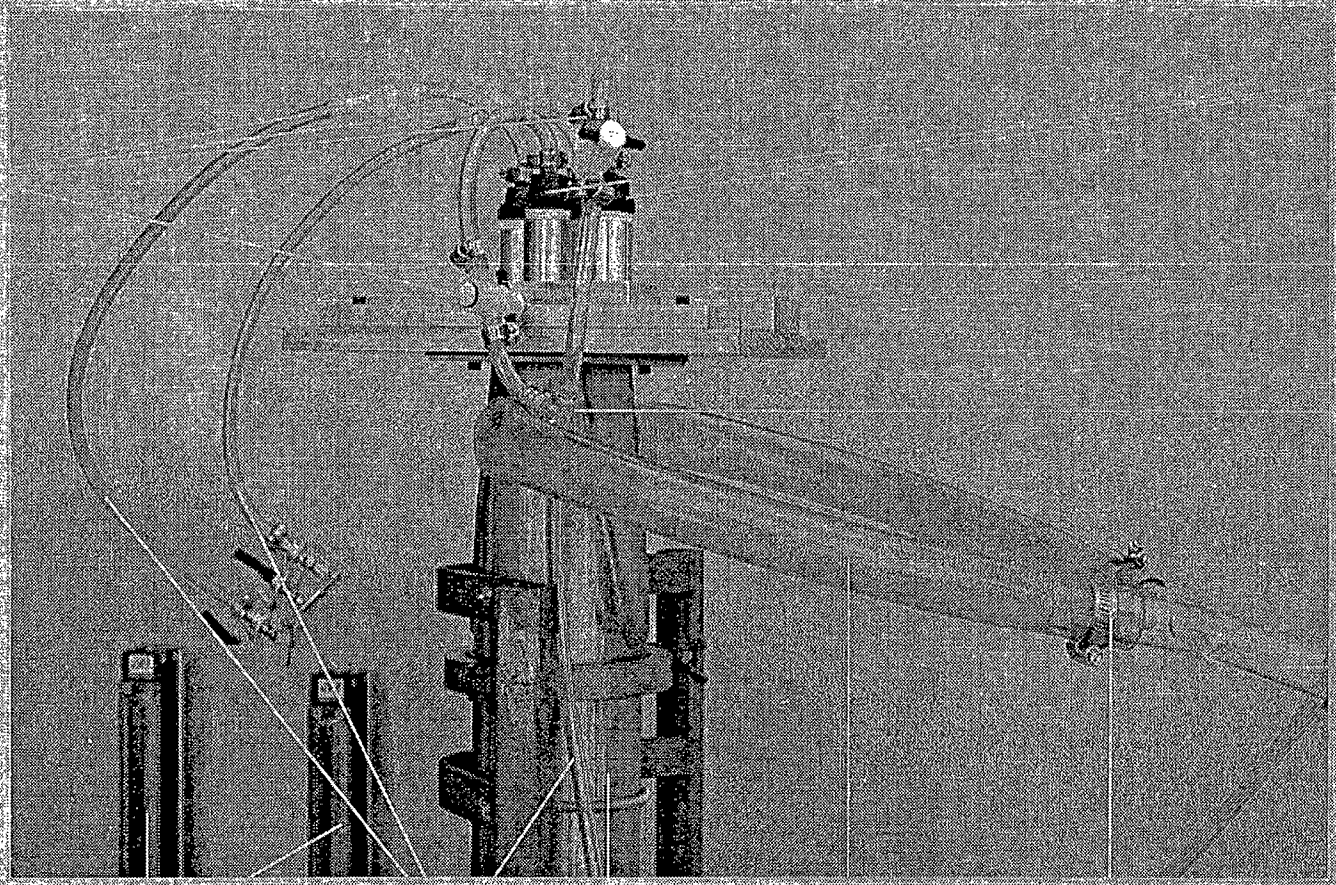
TUBING CONNECTIONS FOR FILTER CASSETTES MOUNTED ON THE ELUTRIATOR OF THE DUST GENERATOR



⊗ Stop Cock Valves

FIGURE A-12

DETAIL OF TUBING CONNECTIONS AT THE TOP OF THE VERTICAL ELUTRIATOR



A.2 THEORY OF OPERATION

A.2.1 The Dynamics of Dust Generation

The dynamics of the release of dust from a sample during a run using the dust generator have been evaluated so that the rate of release and mass of dust in the sample can be derived from measurements of the mass of dust deposited over time on the set of filters collected over the ME opening of the elutriator. Analysis of data obtained from several different types of samples during the pilot study for this method (Berman et al., 1994) indicate that the rate of release of mass from a sample in the dust generator is well described by a first-order rate equation:

$$-dM_s/dt = k*M_s \qquad \text{(A-1)}$$

where:

M_s is the mass of respirable dust remaining in the sample at time "t" (g);

t is the time since the start of the run (s); and

k is the first-order rate constant for the release (s^{-1}).

The minus sign in this equation indicates that mass is lost with time.

Equation A-1 can be integrated to yield:

$$\ln(M_s) = \ln(M_o) - kt \qquad \text{(A-2)}$$

where:

M_o is the mass of respirable dust in the sample at the start of the run (i.e. at time t = 0) (g).

Given that "M_s" can also be expressed as the difference between "M_o" and "M_r" the cumulative mass released up to time "t," Equation A-2 can also be expressed as:

$$\ln(M_o - M_r) = \ln(M_o) - kt \qquad \text{(A-3)}$$

where:

M_r is the cumulative mass released between the start of a run and time "t" (g).

The relationship presented in Equation A-3 indicates that a plot of the natural logarithm of the quantity ($M_o - M_r$) versus time should be a straight line with a slope equal to the rate constant for dust release, k, and an intercept equal to the initial mass of dust in the sample at the start

of the run, M_o. The cumulative mass of dust released from the sample over time, M_r, can be derived from measurements of dust collected on filters during the run. However, because M_o also appears as part of one of the parameters that must be plotted to evaluate the relationship expressed in Equation A-3, the value of M_o must be optimized using regression, as described in Section 11.2 of the main text of this method.

The cumulative mass released from a sample at time "t" during a run, "M_r" is directly proportional to the cumulative mass measured on filters collected during the run:

$$M_r = M_f*(F_s + F_d + F_c)/F_c \quad \text{(A-4)}$$

where:

M_f is the cumulative mass measured on filters collected from filters mounted over the top of the elutriator up to time "t" (g);

F_s is the rate of airflow through the scrubber (cm^3/s);

F_d is the rate of airflow through the IST opening of the elutriator (cm^3/s); and

F_c is the rate of airflow through the ME opening of the elutriator (cm^3/s).

Because F_s and F_c will typically have been set to $0.48*V_v$ and F_d will typically have been set to $0.047*V_v$ during the initial setup of the dust generator (see Section 9.3.4), for most applications, Equation A-4 reduces to:

$$M_r = 2.1*M_f \quad \text{(A-5)}$$

As indicated above, values for M_o must be derived by performing a regression analysis of the relationship described by Equation A-3. This can be accomplished by using any of several commercially available spreadsheet programs (such as, for example, LOTUS™). The procedure to be followed to derive estimates of M_o and k are described in Section 11.2.

A.2.2 The Time Dependence of Dust Collection

As indicated in Section A.2.1 above, the generation of dust from the tumbler is well described by the first order rate equation:

$$-dM_s/dt = k*M_s \quad \text{(A-1)}$$

However, experience gained during the pilot study for this method (Berman et al. 1994) further indicates that the rate of change of M_s is sufficiently slow in most cases such that, for periods of no more than 5 to 10 minutes, M_s can be considered constant. Thus, for

estimating such things as the time required to load individual filters in the dust generator, a simpler form of Equation A-1 can be used (in which M_s is considered constant):

$$\Delta M_s = k*M_s*\Delta t \qquad \text{(A-6)}$$

where:

M_s is still the mass of respirable dust remaining in the sample at time "t" but it is assumed constant over the short interval of time "Δt" (g);

ΔM_s is the mass of respirable dust released from the sample over the short time interval "Δt" (g);

Δt is a relatively short time interval (no more than ten minutes) during which the release of dust is being estimated (s); and

k is still the first-order rate constant for the release (s^{-1}).

Based on Equation A-6, the mass of respirable dust deposited on a filter in the dust generator, call this ΔM_f, is simply the product of the dust released from the sample, ΔM_s, and the fraction of the flow through the dust generator that is also directed through that filter. Thus, for filters collected over the isokinetic sampling tube (the IST opening of the elutriator):

$$\Delta M_f = 0.047* k*M_s*\Delta t \qquad \text{(A-7)}$$

or for filters collected over the ME opening of the elutriator:

$$\Delta M_f = 0.48* k*M_s*\Delta t \qquad \text{(A-8)}$$

The correct value for M_s to be used with Equations A-7 and A-8 is the value estimated by the relationship provided in Equation A-2 of Section A.2.1 where the "t" in Equation A-2 is the time that has elapsed from the beginning of the run to the start of the interval of interest, "Δt" .

NOTE

In deriving Equations A-7 and A-8, it was assumed that air flow within the dust generator was setup as described in Section 9.3.4.

A.2.3 Size Separation Using the Vertical Elutriator

Separation of the respirable fraction of a particulate matrix can be accomplished by exploiting differences in the settling velocities of particles of different sizes when such particles are suspended in either a liquid or gaseous medium. However, air was selected as the medium into which samples would be suspended in this method to avoid changes in the characteristics of asbestos that typically occur when asbestos samples are placed in water[2]. The force on a particle suspended in a moving fluid is given by Stoke's Law (Fuchs, N.A. 1964). When such a particle is suspended in a fluid that is moving upward such that the force of the fluid and the force of gravity on the particle just balance and the particle remains motionless, Stoke's Law indicates that the following relationship holds:

$$4/3*\pi*r^3*d*g = -6*\pi*\eta*r*V_l \qquad \text{(A-9)}$$

where:

- r is the radius of the particle (cm);
- d is the density of the particle (g/cm^3);
- g is the acceleration due to gravity (cm/s^2);
- η is the dynamic viscosity of the fluid (g/cm*s); and
- V_l is the linear velocity of the fluid (cm/s).

The velocity estimated in Equation A-9 is termed the Stoke's velocity, V_s.

By substituting the viscosity of air at room temperature (i.e. 20^o C) and the acceleration due to gravity into Equation A-9, the value of the Stoke's velocity of a particle is estimated as follows:

$$V_s = 1.18*10^6*r^2*d \qquad \text{(A-10)}$$

where "r" is the radius of the particle and "d" is the density of the particle. Because respirable particles are generally defined as those exhibiting an "aerodynamic equivalent diameter" of 10 μm, where an aerodynamic equivalent diameter is the diameter of a particle of unit density that settles at the same rate as the particle of interest, Equation A-10 can be used to find the Stoke's velocity of the largest respirable particles in the elutriator (i.e. by substituting a radius of 5 μm and assuming a density of 1):

$$V_s = 0.295 \text{ cm/s}$$

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[2] It is well documented that the size distribution of asbestos structures in a sample change when such a sample is placed in water. For an overview of such documentation, see Berman and Chatfield (1990). Typically, the number of small fibers and bundles increases and the number and complexity of clusters and matrices decrease when asbestos samples are placed in water.

The Stoke's velocity for a particle is also equal to the velocity of the fluid stream that will just hold a particle motionless against gravity. Because the goal of the dust generator is to capture all particles that are potentially respirable, the velocity of air within the elutriator should be set so that it is just slightly larger (i.e. by 5%) than the Stoke's velocity estimated above (for the largest respirable particles) so that all respirable particles entering the elutriator will be imparted with an upward velocity and will be carried along with the air stream so that they are, ultimately, either deposited on a filter or passed into the scrubber. Therefore, airflow within the dust generator should be set so that the velocity of air within the vertical elutriator is 5% greater than 0.295:

$$V_l = 0.31 \text{ cm/s.}$$

A.3 DUST GENERATOR PROTOTYPE DESIGN FIGURES AND CONSTRUCTION GUIDELINES

The figures of the prototype dust generator that are included in this package are intended to be suggestive and not meant to be followed in exact detail. However, the dimensions of the various cross-sectional areas of different parts of the dust generator, which affect the relative air flows in various places, need to be followed closely for the dust generator to perform as intended. Other design features, such as couplings, clamps, and seals are intended more to be illustrative; alternate designs can be equally effective.

In spite of the low pressure differentials developed during operation in the prototype, performance was found to be very sensitive to leaks, especially leaks occurring in the isokinetic sampler filter mounts. Design features associated with the dust collection system should therefore be selected so as to minimize the potential for leaks in this area. The air flow path in the elutriator of any dust generator that is constructed for use with this method should be tested for leaks when construction is completed and periodically thereafter.

So that dust generator equipment constructed for use with this method will perform adequately, the following requirements must be incorporated into its design:

- the tumbler must be designed to hold a minimum of approximately 100 g of sample (with ample space left over to allow adequate tumbling) so that field homogenization requirements are not compromised. It should also be designed to assure a reasonably long pathlength over which air passes through the sample and baffles (or square corners) should be incorporated to assure adequate tumbling action and thorough mixing of sample and air;

- the pathlength of the elutriator should be a minimum of 10 times its diameter to minimize the possibility of channeling and the diameter should be large enough to assure a cross-sectional area that is at least 10 times that of the tumbler. This latter requirement is to assure adequate air flow in the tumbler that will effect efficient transfer of sample while flow throughout the device is limited to allow flow in the elutriator to be set at 1.05 times the settling velocity of the largest respirable particle of interest;

- the entrance tube and bottom of the elutriator should be shaped so that sufficient air velocities are maintained in this part of the device to assure a smooth transition (with efficient sample transfer) between flow in the tumbler and flow in the elutriator. It is also recommended that the entrance tube to the elutriator be pointed downward as a further hindrance to channeling in the elutriator; and

- filter mounts in the dust collection system need to allow for ready, facile exchange of filters while minimizing the potential for air leaks in this area. A modification incorporated into the prototype device to achieve both requirements was to design aluminum mounts that are tapered such that the shaved bottom half of commercially available filter cassettes fit snugly and can be glued in place (to prevent air leaks at this joint). These can then be fitted with filters and joined to the matching top halves of commercially available cassettes. This feature takes good advantage of the fit between the two plastic halves of a commercial filter cassette, which are designed to join with minimal leakage while allowing for rapid exchange of filters. The bottom half of the aluminum filter mounts are sealed into the slide mechanisms on the prototype apparatus with o-rings.

The following figures are included in this package:

- Figure A-13 indicates the overall assembly of the prototype dust generator;

- Figure A-14 indicates the details of the prototype tumbler assembly;

- Figure A-15 indicates the details of the prototype vertical elutriator;

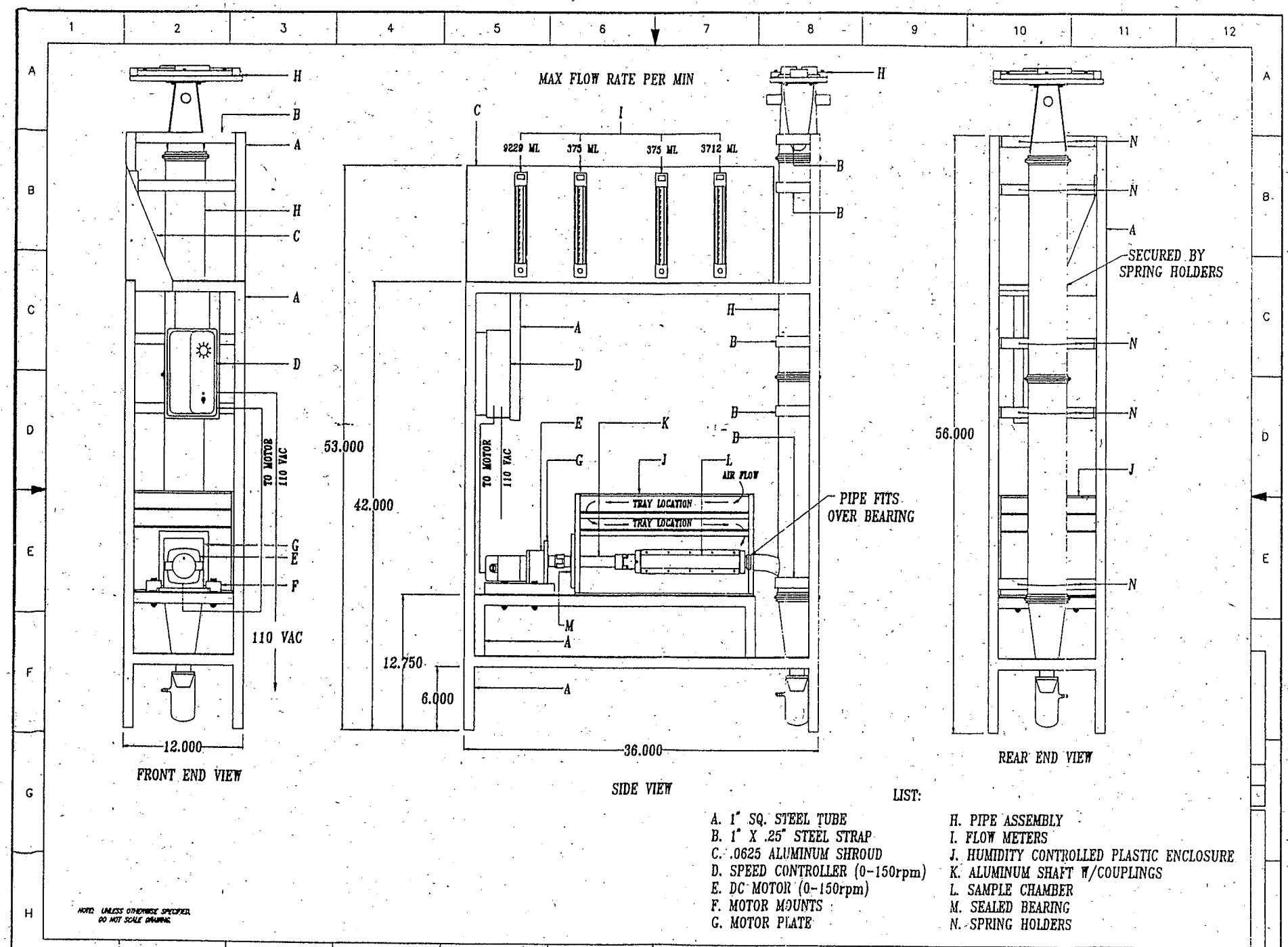
- Figure A-16 is a vertical cross-section of the top of the prototype vertical elutriator indicating the relationship between the elutriator openings, the isokinetic sampling tube, and the slide mechanisms of the dust collection system;

- Figure A-17 indicates the details of the isokinetic sampling tube;

- Figure A-18 indicates the details of the prototype slide mechanism of the dust collection assembly; and

- Figure A-19 indicates the details of the prototype filter cassette mounts.

FIGURE A-13 OVERALL PROTOTYPE DUST GENERATOR ASSEMBLY



LIST:

A. 1" SQ. STEEL TUBE
B. 1" X .25" STEEL STRAP
C. .0625 ALUMINUM SHROUD
D. SPEED CONTROLLER (0-150rpm)
E. DC MOTOR (0-150rpm)
F. MOTOR MOUNTS
G. MOTOR PLATE
H. PIPE ASSEMBLY
I. FLOW METERS
J. HUMIDITY CONTROLLED PLASTIC ENCLOSURE
K. ALUMINUM SHAFT W/COUPLINGS
L. SAMPLE CHAMBER
M. SEALED BEARING
N. SPRING HOLDERS

FIGURE A-14 PROTOTYPE TUMBLER ASSEMBLY DETAIL

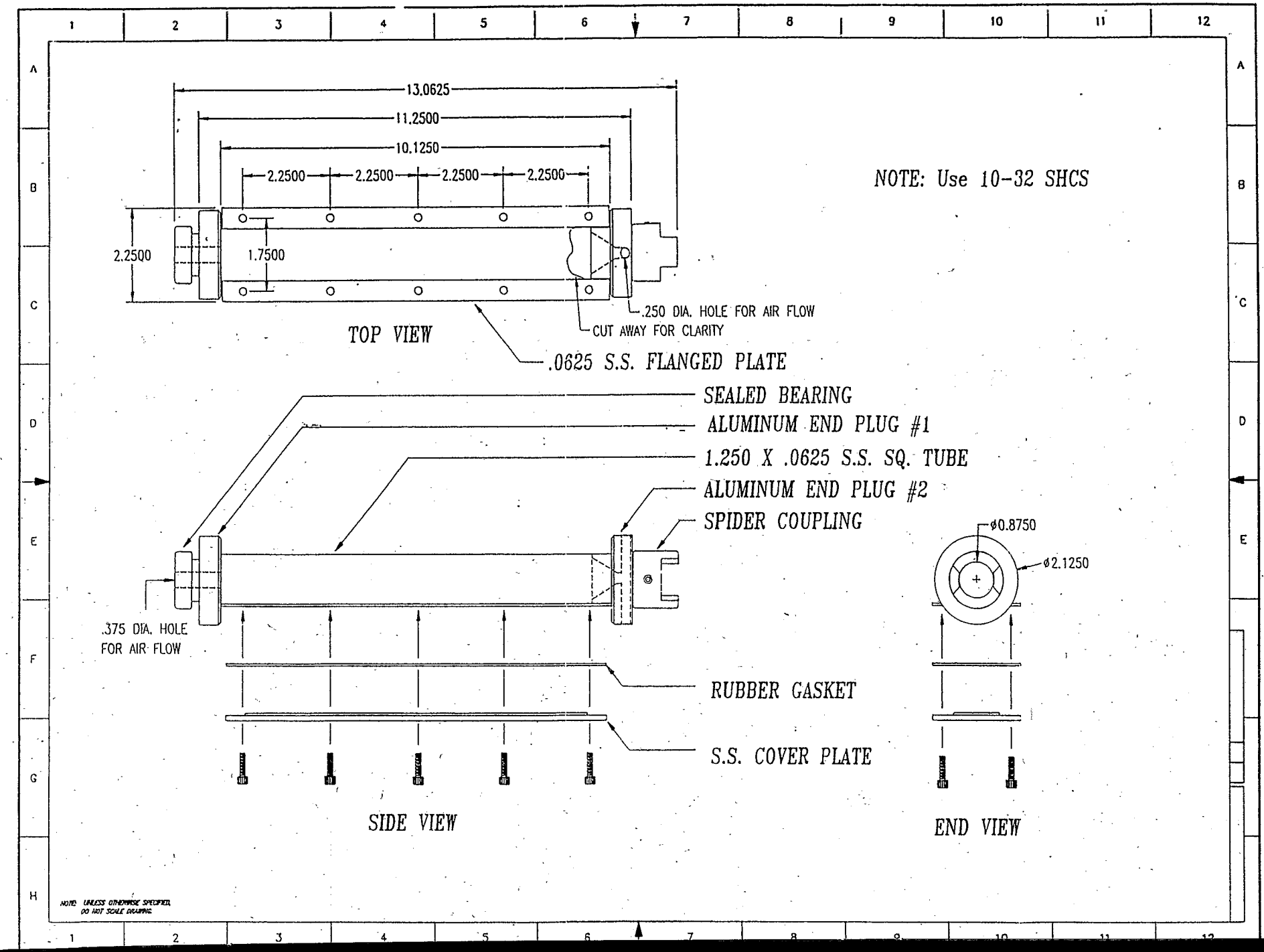


FIGURE A-15 PROTOTYPE VERTICAL ELUTRIATOR DETAIL

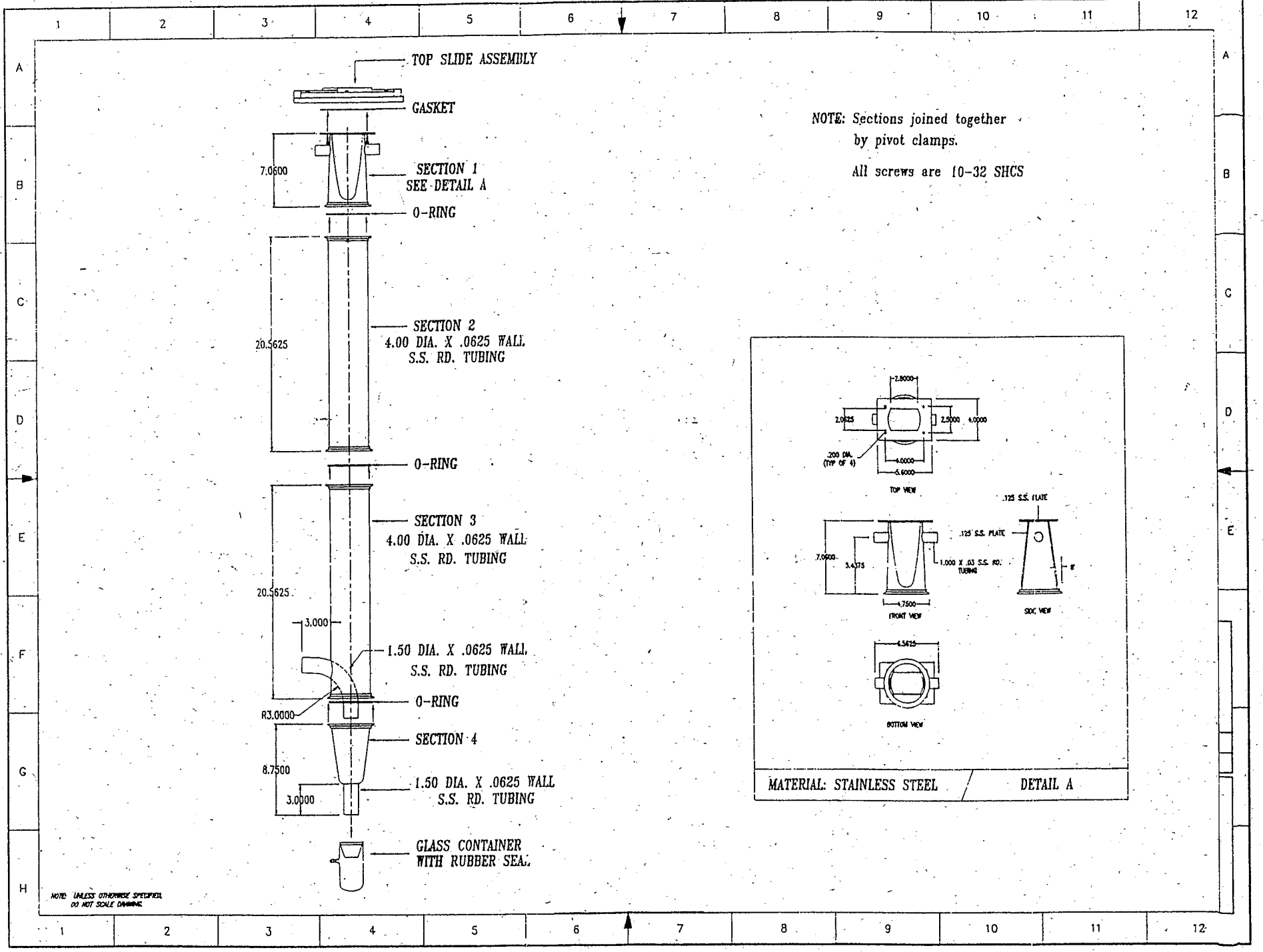


FIGURE A-16

SECTION OF ELUTRIATOR TOP WITH EXTENSION TUBE

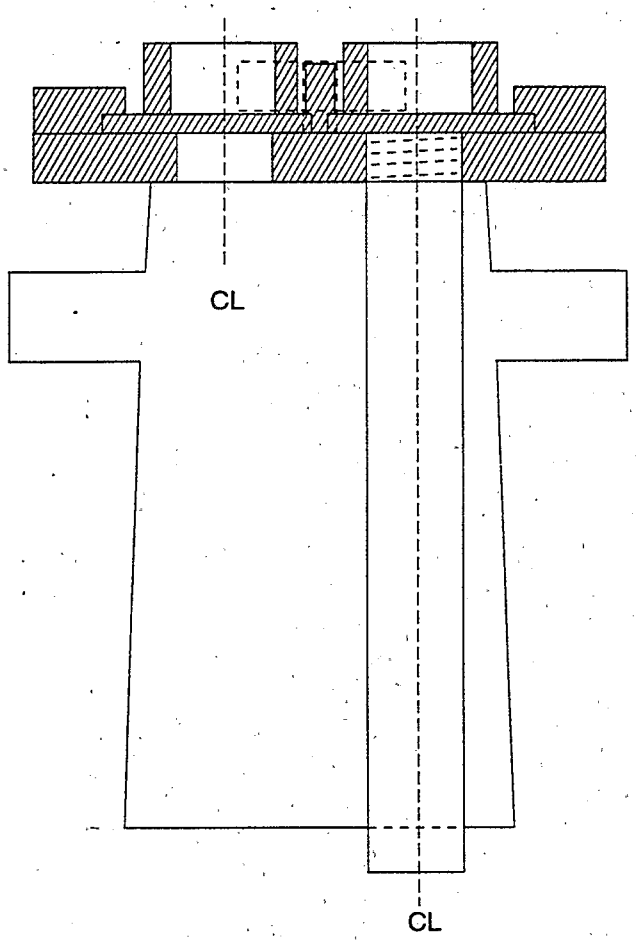


FIGURE A-17

LONGITUDINAL & TRANSVERSE SECTIONS OF ISOKINETIC SAMPLING TUBE

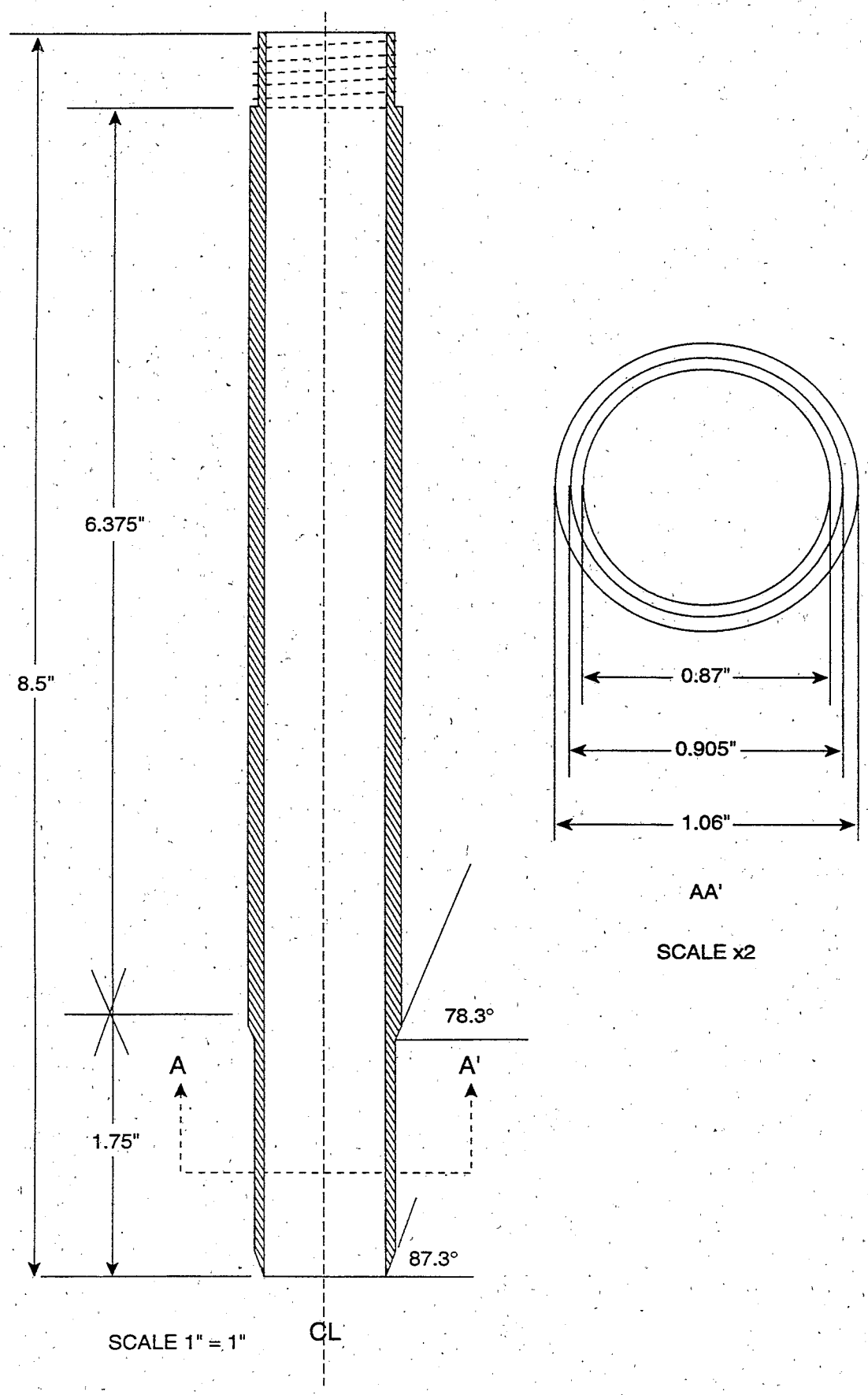
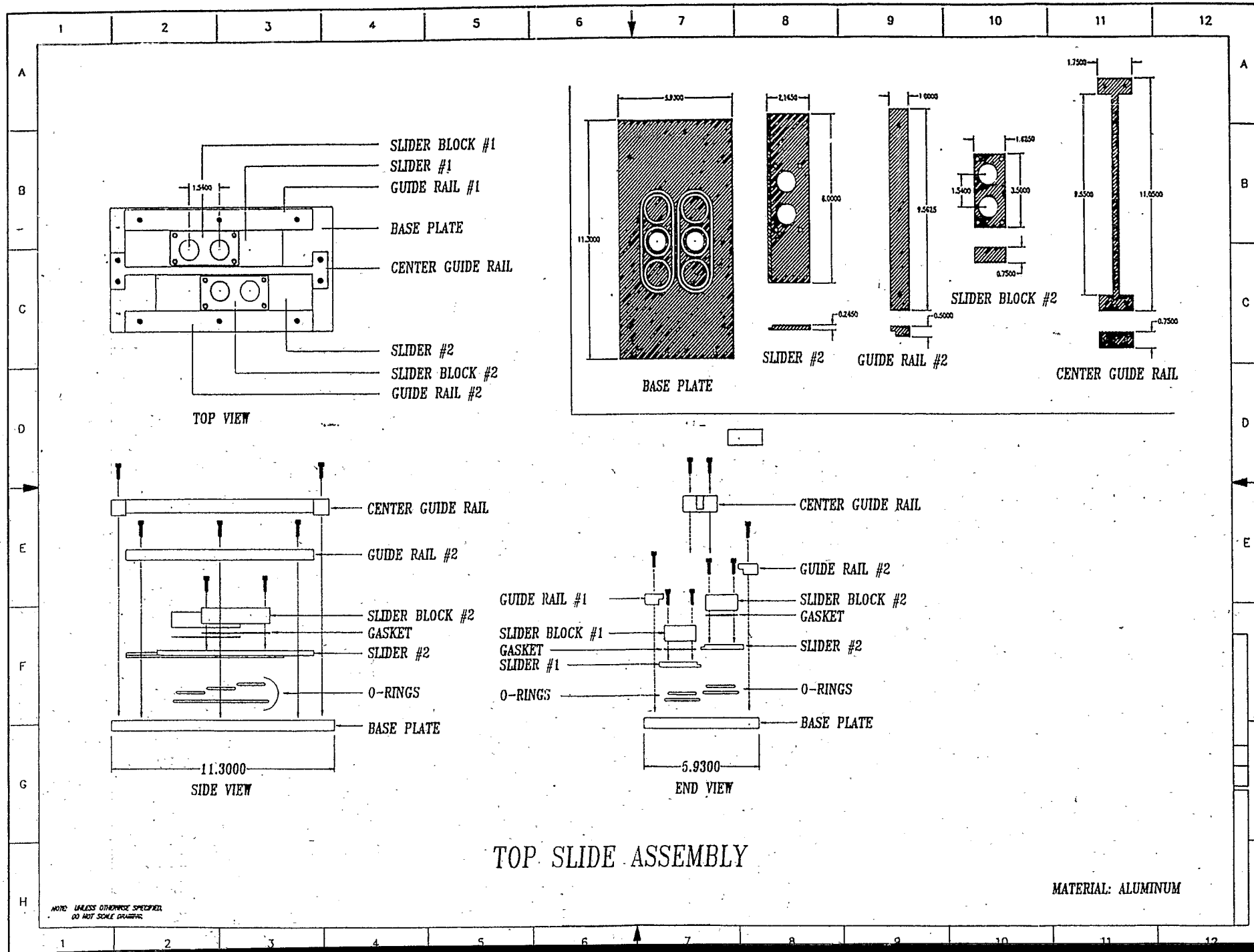


FIGURE A-18 PROTOTYPE SLIDE MECHANISM DETAIL



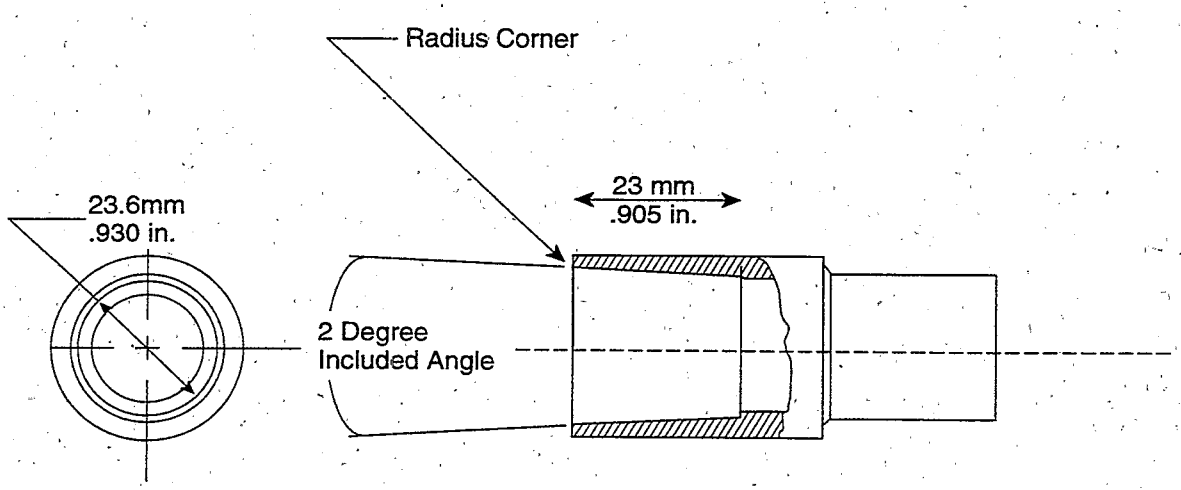
TOP SLIDE ASSEMBLY

MATERIAL: ALUMINUM

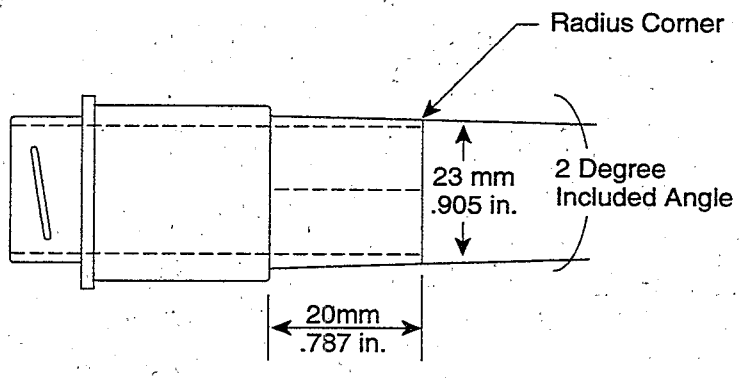
NOTE: UNLESS OTHERWISE SPECIFIED, DO NOT SCALE DRAWING

FIGURE A-19

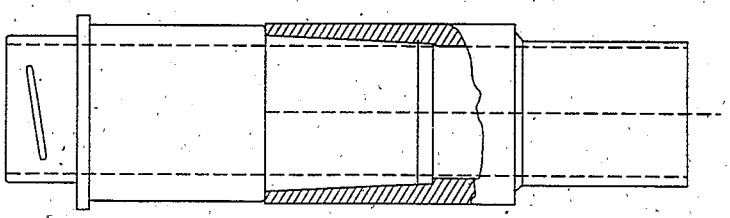
DETAIL OF PROTOTYPE FILTER CASSETTE MOUNTS



ALUMINUM CASSETTE HOLDER, TOP MODIFIED WITH INTERNAL TAPER AS SHOWN



25MM MILLIPORE CASSETTE, BOTTOM MODIFIED WITH EXTERNAL TAPER AS SHOWN



CASSETTE AND HOLDER SHOWN JOINED TO CREATE A TAPER LOCK SEAL

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